Programme Area: Bioenergy

Project: Characterisation of Feedstocks

Title: D12 Final report on investigations into the effect of harvest time and variety on willow SRC and the effect of harvest time and storage method on Miscanthus quality

Abstract:

The primary objective of this 2015/16/17 Project was to provide an understanding of UK produced biomass properties, how these vary and what causes this variability.

This is a copy of the slide set presented by the Forest Research led team covering the findings from the whole of the Characterisation of Feedstocks Project. This set of 71 slides was presented at the ETI on 16th March 2017.

Context:

The Characterisation of Feedstocks project provides an understanding of UK produced 2nd generation energy biomass properties, how these vary and what causes this variability. In this project, several types of UK-grown biomass, produced under varying conditions, were sampled. The biomass sampled included Miscanthus, Short Rotation Forestry (SRF) and Short Rotation Coppice (SRC) Willow. The samples were tested to an agreed schedule in an accredited laboratory. The results were analysed against the planting, growing, harvesting and storage conditions (i.e. the provenance) to understand what impacts different production and storage methods have on the biomass properties. The main outcome of this project is a better understanding of the key characteristics of UK biomass feedstocks (focusing on second generation) relevant in downstream energy conversion applications, and how these characteristics vary by provenance.

ETI Project Code: BI2010

Project Title: Characterisation of Biomass Feedstocks

Deliverable title: Final report on investigations into the effect of harvest time and variety on willow SRC and the effect of harvest time and storage method on *Miscanthus* quality

Reference: D12

Participant lead: Forest Research

Other participant: Uniper Technologies Ltd

Submission date: 9th March 2017

Version number: v2.2

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Executive Summary

Within the UK, the installation of dedicated biomass power plants and the conversion of existing coal plants to use biomass, either in dedicated plants or co-fired with coal, has dramatically increased the demand for biomass feedstocks. At present home-grown output is significantly less than the demand, creating the opportunity for UK land-owners to supply this new market. Despite this, the level of understanding of biomass crops in the UK is still rather general. In particular, there is limited understanding of the variability in feedstock properties and a lack of recognition that differences in various properties can have a significant effect on the subsequent conversion to power and/or heat. Across all scales of use, feedstock quality is critically important in order to optimise plant performance, safeguard the environment, and maximise the financial benefits of the project.

The specific objectives of this deliverable (D12), which covered all the investigations of Phase 2, were to:

- Study 5: Identify the impact of harvest time on the feedstock qualities of UK-produced *Miscanthus*. In addition there is an opportunity to compare feedstock characteristics in spring 2016 with those of samples collected in spring 2015 under Phase 1 of the contract.
- Study 6: Identify the impact of harvest time on the feedstock qualities of UK-produced willow short rotation coppice (SRC). In addition there is an opportunity to compare feedstock characteristics in spring 2016 with those of samples collected in spring 2015 under Phase 1 of the contract.
- Study 7: Identify the impact of willow SRC varieties on willow SRC crop qualities
- Study 8: Identify the impact of these storage types on *Miscanthus* properties with time

Studies 1-4 formed Phase 1 of this Project and are reported in Deliverable D6.

Yields of *Miscanthus* and willow SRC have been studied extensively so typical averages and ranges are established. Consequently the project focusses on feedstock quality and no yield data were measured.

The final contract deliverable (D13) summarises the results of both Phase 1 and Phase 2, discusses the representativeness of the findings, and draws out the implications for growers and end-users of the four feedstocks investigated in the contract. D13 tabulates and graphs the results for each of the key feedstock characteristics.

Many of the *Miscanthus* characteristics of crops grown at six sites ranging from Lincoln to south west England changed significantly through time. The majority of values - moisture content as received (ar), ash as percent of dry fuel (d), carbon on a dry ash-free basis (DAF), chlorine (DAF), molybdenum (d), zinc (d), bromine (d), phosphorus (d), silicon (d), and calcium (d) - decreased over the sampling duration of early November 2015 to late May 2016. Of these, many showed a decreasing trend throughout the period, i.e. during the time both as a standing crop (November 2015 to March 2016) and also as the crop was harvested commercially and left on the stubble until baling (until late May at the longest). There were however interesting variants of this general decreasing trend, in particular nitrogen (DAF), which increased slightly in early April. A smaller number of variables - net calorific value (ar) and volatile matter (DAF) - increased between November 2015 and June 2016 and in one

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case (sodium (d)) levels increased through the winter and early spring but then decreased again.

The findings were consistent with the literature and confirm a general decrease through late autumn, winter and early spring in moisture content (ar), ash (d), carbon (DAF), nitrogen (DAF), chlorine (DAF), molybdenum (d), zinc (d), bromine (d), phosphorus (d), silicon (d), and calcium (d) accompanied by an increase over the same period in net calorific value (ar), volatile matter (DAF), and sodium (d). Several sites, mainly those in south west England, showed a previously unreported pattern for *Miscanthus* of increasing nitrogen (DAF) in the late spring which may be associated with a resumption of growth in stems.

A comparison between the same calendar dates in 2015 and 2016 suggested that the *Miscanthus* was drier in 2015. For a given site some characteristics, e.g. chlorine, were lower while calcium was higher, date for date, in 2015. With these exceptions the levels in the assessed characteristics were generally similar in the two years up to the point of harvesting and the trends were also similar in the two years. Changes between harvesting and the pre-baling sample were less consistent across sites and years. We hypothesised that these differences were related to differences in rainfall at the individual sites between harvesting and baling. It was not possible however to identify any unequivocal, simplistic correlations between either rainfall totals or intensity and changes in feedstock characteristics between the time of commercial harvesting and baling.

Only a few characteristics of willow SRC grown at six sites from north west to southern England showed statistically significant differences across the three simulated harvesting times (mid-November 2015, mid-January 2016 and mid-March 2016), with the majority showing no difference. For the characteristics that did change – GCV (DAF), chromium (d), $CaCO_3$ normalised ash (na), K_2O (na), and P_2O_5 (na) - several patterns were evident. Gross calorific values at all sites fell between mid-November and mid-January; by mid-March the GCV had increased again to reach levels mid-way between the mid-November and mid-January values. A comparison between the analysis obtained in 2015 and 2016 suggested that the observed increase in GCV continued through April and May. It appeared that the levels of chromium increased throughout the period. Other trends observed in spring 2016 were not corroborated by spring 2015 data, with the possible exception of P_2O_5, which seemed to decline slightly between mid-January and mid-March 2016 and also between mid-February and early May 2015.

There are several possible reasons for the limited number of significant differences observed in willow SRC, especially in relation to *Miscanthus*. For example, direct comparison at a given site was not possible for willow SRC, there was a wider geographical range of willow SRC sites which resulted in a wider range of climate zones and soil types, there were several different varieties within a willow SRC site and different varietal mixes at different sites, and lastly there were fewer willow SRC sampling occasions. On the basis of published literature the results are not unexpected. There are few external papers to support significant seasonal changes in proximate and ultimate fuel properties or ash-forming elements (only changes in GCV were significant in the present study) and although willows are well known as bio-accumulators of trace elements and heavy metals, the soils at the sites studied were very low in concentrations of both.

Considered as a whole, our results suggest that to maximise *Miscanthus* quality, harvesting should be delayed until at least the beginning of March, with chlorine and ash a particular concern if harvesting is brought forward, which also risks losing the advantages of low

moisture content and higher NCV. In the case of willow SRC, our results suggest that have greater flexibility over harvesting times. This window however should be limited to after leaf fall through to bud burst because inclusion of leaf material risks raising the moisture content, ash, nitrogen, sulphur and chlorine levels considerably. To quote Heaton *et al.* (2009): *"*Put succinctly: there is a trade-off to consider when harvesting perennial biomass crops: harvest too late and yield declines, harvest too early and risk higher mineral contents, particularly nitrogen (N).*"*

There was a certain degree of consistency in willow SRC variety properties across sites from Northern Ireland to southern England, with approximately 40% of the parameters analysed showing statistically consistent rankings for the varieties tested (Endurance, Nimrod, Resolution, Sven, Terra Nova, and Tora). The most highly significant differences ($P<0.01$) in rankings were in moisture, net calorific value, carbon, nitrogen and $CaCO_3$. For example, Endurance was consistently the lowest in terms of moisture content, with Tora, Resolution and Sven in the mid-range, and Terra Nova and Nimrod generally having the highest moisture content. Resolution generally had low nitrogen concentrations whereas Nimrod, Terra Nova and Endurance generally had high nitrogen levels with Sven being intermediate.

In addition, differences in the ranking of gross calorific value, copper, calcium, magnesium, manganese, K_2O, MgO and Na_2O were significant with *$P<0.05$ and >0.01*. For example, Nimrod had consistently high gross calorific values, Tora had consistently low levels of copper, while Resolution, followed by Sven, had consistently low levels of calcium.

Considering the results as a whole, there was evidence of consistent differences across a wide geographical range in approximately 40% of the important parameters analysed but no variety combined the best ranking in all parameters across all sites. Conversely our results suggest that for the majority of parameters, there was not a consistent ranking. Volatile matter, sulphur and chlorine content for example did not show consistent rankings and neither did the alkali index.

These results are based (quite deliberately) on a snapshot in time and the impact of seasonality should be considered. Study 6 showed that most of the parameters fluctuated markedly over the three sampling times therefore the findings about the consistency of rankings should be applied to a wider time frame only with considerable caution.

The experimental *Miscanthus* storage treatments were representative of the majority of commercial systems currently used in Britain in terms of the storage method and duration. This study makes available for the first time evidence on the impact of storage type and duration on important feedstock characteristics. The findings can be grouped into three:

- ca 43% of the feedstock characteristics tested were not significantly affected by storage;
- another 43% were affected by storage but there was no influence of the method of storage; the majority of these changes decreased fuel quality; and
- in the remaining 14% of analysed feedstock characteristics, which included ash, nitrogen, sulphur, zinc, bromine and calcium, storage treatments did have a significant influence; again the majority of these changes decreased fuel quality.

Although the method of storage did not affect levels of potassium, the oxide of potassium or the alkali index, these important characteristics were all significantly lower after storage, which represented improvements in condition.

From a practical point of view, some period of on-farm storage is likely to be needed so the question becomes: what can be done to minimise the deterioration? Although industry advice is that indoor storage with a dry floor is preferred, our study indicated that sulphur and zinc levels increased more during indoor storage compared to outdoor storage. Furthermore ash values were much higher in the covered storage in the samples after one month's storage. Nitrogen decreased (improved) more in the outdoor uncovered bales but this type of storage also increased calcium more than the other treatments. These results suggest that no single type of storage is likely to minimise the deterioration in all aspects of feedstock quality and the choice of storage type is more likely to be dictated by what type of storage is available and perhaps the contamination risk on the farm, especially in the absence of any price differential linked to quality.

Although the 5-month storage duration represented a typical operational situation, the questionnaire showed that both much shorter and longer periods may be used to fit with work patterns on the farm and market demands. Since this project demonstrated major changes in many aspects of *Miscanthus* quality during storage and also that the storage method and duration could be influential, these findings should be considered carefully by the sector.

Contents

1   Introduction

1.1   Background and context

At a global scale, the use of biomass as a source of energy has transformed in the last two decades, from widespread utilisation within a local geographic area for cooking and heating, to use for power generation at a large scale and combined heat and power generation at a medium scale. This change is largely in response to policy and financial drivers to limit CO_2 emissions from fossil fuels, with biomass combustion generally considered to be low carbon, or even CO_2 neutral (although account must be taken of the sustainability of the supply and upstream emissions from production and transport). As a result of the biomass tonnages required for large-scale industrial operations, global trading of biomass feedstocks is now well established. The more traditional use for heat persists and has even grown in some developed countries, due to a variety of reasons, including environmental, financial and aesthetic reasons, as well as a lack of alternative sources of heating in some locations.

Across all scales of use, feedstock quality and properties are critically important in order to optimise plant performance, safeguard the environment and maximise the financial returns of the project. From a review of the international literature, it is clear that harvesting time affects the properties of the perennial grass *Miscanthus x giganteus* and willow short rotation coppice (SRC) as a result of both internal mobilisation of nutrients from above-ground growth into storage rhizomes and woody roots respectively at the end of the growing season and of leaf fall in autumn. Where literature relates to countries close to Britain geographically the location is specified.

Delaying harvest overwinter tends to reduce the overall yield – as just one example we cite Lewandowski and Heinz (2003) who found that bioenergy yields (in terms of GJ ha-1) of *Miscanthus* crops in three sites in southern Germany decreased by 14–15% between December and February and by a further 13% between February and March. Himken *et al.* (1997), Clifton-Brown and Lewandowski (2002) recorded substantial reductions in yield of up to 30%.

Although yield is a key determinant of the economic viability of biomass crop, this project focusses on the changes in composition. Seasonal changes in a wide range of feedstock characteristics have been reported for *Miscanthus,* For example, N, P, K and Mg decreased during summer and autumn, whereas Ca and Na concentrations increased in *Miscanthus* crops which had grown at sites with different levels of soil Cd, Pb and Zn (Nsanganwimana *et al.,* 2016). The continuing changes through autumn and winter have major implications for both the quantity and quality of the harvest. Delaying harvesting from the autumn to the following spring improved the quality of the harvested biomass by a reduction in N, K and Cl as well as a reduction in moisture content through drying (Himken *et al.,* 1997; Heaton *et al.,* 2009*;* Di Nassi *et al.,* 2011). Lewandowski *et al.* (2003) reported that the reduction in yield between December and March was accompanied by a significant decrease in water content and in the ash, nitrogen, chloride and sulphur concentrations of the harvested biomass. Heaton *et al.* (2009) found that there was a major reduction in N concentration between June and December but then little additional change over the following months to February/March. In a 5-year study in Carlow (Ireland), Finnan and Burke (2014) found that N mobilisation between October and February was small and most of the N decrease over this period was due to loss of leaves following abscission. Jensen *et al.* (2016) examined the effect of

senescence and flowering time of 16 different *Miscanthus* genotypes (including *Miscanthus x giganteus)* over summer, autumn and the following spring in trials grown near Aberwystwyth, Wales. Contrary to expectation they found that five genotypes, including *Miscanthus x giganteus,* did not show lower levels of N, P and K in spring (February) than in the previous autumn (October); moreover in two of these genotypes, again including *Miscanthus x giganteus,* Cl did not decrease over winter. In a study that spanned 5 Degrees of latitude, contrasting soil types and land capability classes, Arundale *et al.* (2015) reported that delaying harvest from October to December decreased the proportion of hemicellulose, acetyl groups and ash but increased the cellulose and lignin at statistically significant levels.

In addition to changes associated with the seasonal growth pattern of *Miscanthus*, annual variation should be considered. Christian *et al.* (2008) followed the growth, yield and mineral content of *Miscanthus x giganteus* over 14 successive harvests and found that the total production differed by only 5%. Based on a modelling study of European *Miscanthus x giganteus* crops, Miguez *et al.* (2008) concluded that the growth curves of dry matter production as a function of thermal time, i.e. accumulated daily average temperature over the growing season, were relatively stable. Closer to home, Richter *et al.* (2008) developed an empirical model of *Miscanthus* yields in 14 arable sites across the UK over 3 years, which showed that available soil water, air temperature and precipitation were the main factors explaining the variation in yield. Although there are some reports of differences in yield from year to year there are almost no reports of differences in feedstock characteristics. A study by Baxter *et al.* (2014) of the impact of various N and K fertiliser combinations on overwinter *Miscanthus* properties in the 3rd and 4th year after planting, found some similar responses in the two years: K_2O content generally fell but from February onwards increased slightly, whereas SiO_2 generally increased but from February onwards decreased slightly. By contrast the patterns of calorific value, nitrogen and ash differed in the two years. For example, there was no pronounced trend in ash in the 3rd year crop but a decrease in the 4th year crop; calorific value tended to increase throughout the sample period (early November to mid-March) of the 3rd year crop but in the 4th year this increase was less evident and seemed to occur later in the period. In summary, although the literature indicates some general trends in properties through the autumn, winter and into spring, these are not entirely reliable from year to year even at the same site and differ from one property to another.

Since ideal crop characteristics at harvest for power and heat end uses are low N which gives low NO_x emissions, low K and Cl to reduce boiler corrosion, as well as low moisture to give higher NCV, there is clearly a practical value in comparing the impact of harvest time in a range of commercial *Miscanthus x giganteus* crops. Mos *et al.* (2013) have shown that harvest time and senescence of *Miscanthus x giganteus* affected bio-oil quality and stability following fast pyrolysis so a greater understanding of the impact of harvest time is important for a wide range of conversion technologies. Moreover there are practical implications for *Miscanthus* growers, e.g. low N and P removals in the harvested biomass may reduce subsequent fertiliser inputs, and low moisture may reduce spoilage and transportation costs. High moisture content on the other hand may have a negative impact on the value of the crop to the end user so the grower may be paid less.

In willow SRC, the seasonal pattern of elemental concentrations differed from one element to another. Between 40% of the N and about 60% of the P was withdrawn from willow SRC leaves prior to abscission and stored mainly in the below-ground organs, however, there were no seasonal trends or translocation from senescing leaves for Ca and Mg and translocation of

K and S were observed only in plants grown under a low nutrient regime (Von Fricks *et al.*, 2001). By contrast, for a range of willow SRC species, concentrations of Cd and Zn in leaves, wood and bark increased towards the end of the growing season (Mertens *et al., 2006); likewise* Migeon *et al.* (2009) reported seasonal variation in Cd, Zn, and Pb in a range of woody species including willows.

Put succinctly: there is a tradeoff to consider when harvesting perennial biomass crops: harvest too late and yield declines, harvest too early and risk higher mineral contents, particularly nitrogen (Heaton *et al.* 2009).

Willow species have been interbred for many decades, and the resultant crosses show a wide range of growth rates, stem and leaf forms, resistance to pests and diseases, as well as physiological differences. In a comparison of yield, resistance to pest and disease, biomass composition and wood density on two contrasting sites, Serapiglia *et al.* (2013) found consistent genotypic differences in yield which were associated with susceptibility to rust and beetle damage.

Tharakan *et al.* (2005) assessed morphological differences among 30 willow SRC clones. They reported that one set of clones was characterised by a large number of small diameter stems, relatively low leaf area index and specific leaf area but high foliar nitrogen and wood specific gravity. By contrast, the other set was characterised by a small number of large diameter stems, high leaf area index and specific leaf areas but low foliar nitrogen and wood specific gravity. Brereton *et al.* (2014) investigated the N dynamics during growth and onset of winter dormancy in 14 willow SRC genotypes by measuring elemental analysis and ^{15}N isotopic labelling in June, August and October; they observed genotype-specific variation for all the traits they measured. It is not surprising therefore that these differences have the potential to affect conversion outcomes. In a comparison of seven willow SRC varieties grown in a moderate European climate, Krzyzaniak *et al.* (2014) found that the content and yield of cellulose and hemicellulose in two clones of *Salix viminalis* makes them particularly useful for an integrated multi-product bio-refinery based on lignocellulosic feedstocks. In a study of willow SRC varieties in England, Gudka (2012) found differences in cellulose, hemicellulose and lignin content, calorific value and both total ash content and the percentage of different oxides in the ash were observed. In general varieties with the lowest lignin had lowest ash with % CaO being negatively correlated with K_2O.

As with *Miscanthus,* ideal crop characteristics at harvest for power and heat end uses are low N to reduce NO_x emissions, low P to reduce slagging and fouling, and low K and Cl to reduce boiler corrosion so there is clearly a practical value in understanding varietal differences, particularly if these differences tend to be stable from site to site. Likewise low N and P removals in the harvested biomass may reduce subsequent fertiliser inputs so there are possible beneficial implications for willow SRC growers. Where consistent varietal differences exist, they are likely to be linked to genetically determined traits, such as the number and dimensions of stems, bark thickness, the root:shoot ratio, the root surface area and the distribution of roots.

Most of the guidance on storage regimes of *Miscanthus* is provided in fact sheets from management companies and there are limited data in the scientific literature. There is however consensus that moisture content should be below 16 % at the start of storage (pers. comm. M Mos 2015) and at temperatures below 15 °C (Sustainable Energy Authority Ireland, 2015). No information was found on the change in feedstock characteristics during storage. Greenhalf *et al.* (2013) investigated the influence of harvest and storage on the properties of *Miscanthus*

but round bales were used therefore the results are not directly relevant to the storage of rectangular Heston bales.

1.2 **Objectives**

The overall purpose of this deliverable (D12) is to inform the ETI on the variability in feedstock properties of UK-produced energy biomass types. The specific objectives stated in the contract are to:

- Identify the impact of harvest time on UK-produced *Miscanthus* and willow SRC crop qualities. Uncertainties are to be identified.
- Identify the impact of willow SRC varieties on willow SRC crop qualities. Uncertainties are to be identified.
- Identify the common methods used for storing *Miscanthus*
- Identify the impact of these storage types on *Miscanthus* properties with time

In addition there is an opportunity to compare feedstock characteristics in spring 2016 with those of samples collected in spring 2015 under Phase 1 of the contract.

Note that the final contract deliverable (D13) summarises the results of both Phase 1 and Phase 2, discusses the representativeness of the findings, and draws out the implications for growers and end-users of the four feedstocks investigated in the contract. D13 tabulates and graphs the results for each of the key feedstock characteristics.

1.3 **Hypotheses**

The following hypotheses were set out in the contract:

1. Harvest time will affect the fuel properties and/or composition of *Miscanthus* and willow SRC.
2. The feedstock characteristics of *Miscanthus* and willow SRC will differ from one year to the next at a given site.
3. The feedstock characteristics of willow SRC varieties will differ from one variety to another in a consistent manner from one location to another.
4. The fuel properties and/or composition of *Miscanthus* are influenced by the storage method and duration.

2   Methodology

2.1   Sampling and analysis

The aims of the field sampling and laboratory analyses were to maximise consistency and repeatability while minimising contamination, such that the variability of the data was a true reflection of the variability of the feedstock.

Sampling of both soil and feedstocks has been previously described in D2 (Appendices 5 to 12); flow charts in D2 (Appendix 13) described the process for sample collection and dispatch. Standard operating procedures (SOPs) for sampling were developed and are presented in Table 3 of D2. Fuel analysis was primarily undertaken in the ISO17025-accredited internal laboratories of Uniper Technologies, following standard procedures as listed in Table 2 of D2, although ash fusion temperatures were sub-contracted to external ISO17025 accredited laboratories, as were soil analyses. Flow charts in D2 Appendix 14 described the laboratory process for sample preparation and testing.

Because the objectives in Phase 2 had a particular emphasis on the comparison through time, consistency and repeatability were of utmost importance therefore the protocols for both field sampling and laboratory assessments were identical with those in Phase 1 and were followed rigorously at all harvest and storage times.

2.2   Study overview

2.2.1   Study 5: The impact of harvest time on the feedstock characteristics of *Miscanthus*

Six commercial *Miscanthus* sites were selected from the twelve sites used in Phase 1 (see Figure 2-1). Since the objective was to examine the impact of harvest time on the inherent properties of the crop, we selected three sites from three light and three medium soils because we expected soil type to be a significant influence on crop properties. Soil samples were not collected from the sites in Phase 2 since they had already been sampled in Phase 1. For the same reason, sites were sought from within only one climate zone (warm dry) in an attempt to limit the differences from one sampling time to the next due to climate.

On three occasions covering the autumn and winter (early November 2015, early January 2016 and the first half of March 2016), samples were collected from standing crops at the normal cutting height to simulate commercial harvesting. At each site, samples were collected on two further occasions linked to normal commercial practice – immediately after the crop was cut as part of the commercial harvest, and just before the crop was baled. Summary details are provided in Table 2-1 and full details are included in the database D9. As described fully in D2, on each harvesting date 10 representative samples were collected from each site. The 10 samples were bulked, thoroughly mixed and sub-sampled to give a representative sample for each site and harvesting time.

Figure 2-1: **Map showing the location of *Miscanthus* sites used in Study 5 (marked with a X) and Study 8 (marked with a +) in Phase 2 in relation to the sites used in Phase 1.**

Misc. = *Miscanthus*; WD = Warm/dry climate zone; WM = Warm/moist climate zone; IFV = in-field variation from Phase 1; V1 = Study 5; V4 = Study 8 from Phase 2.
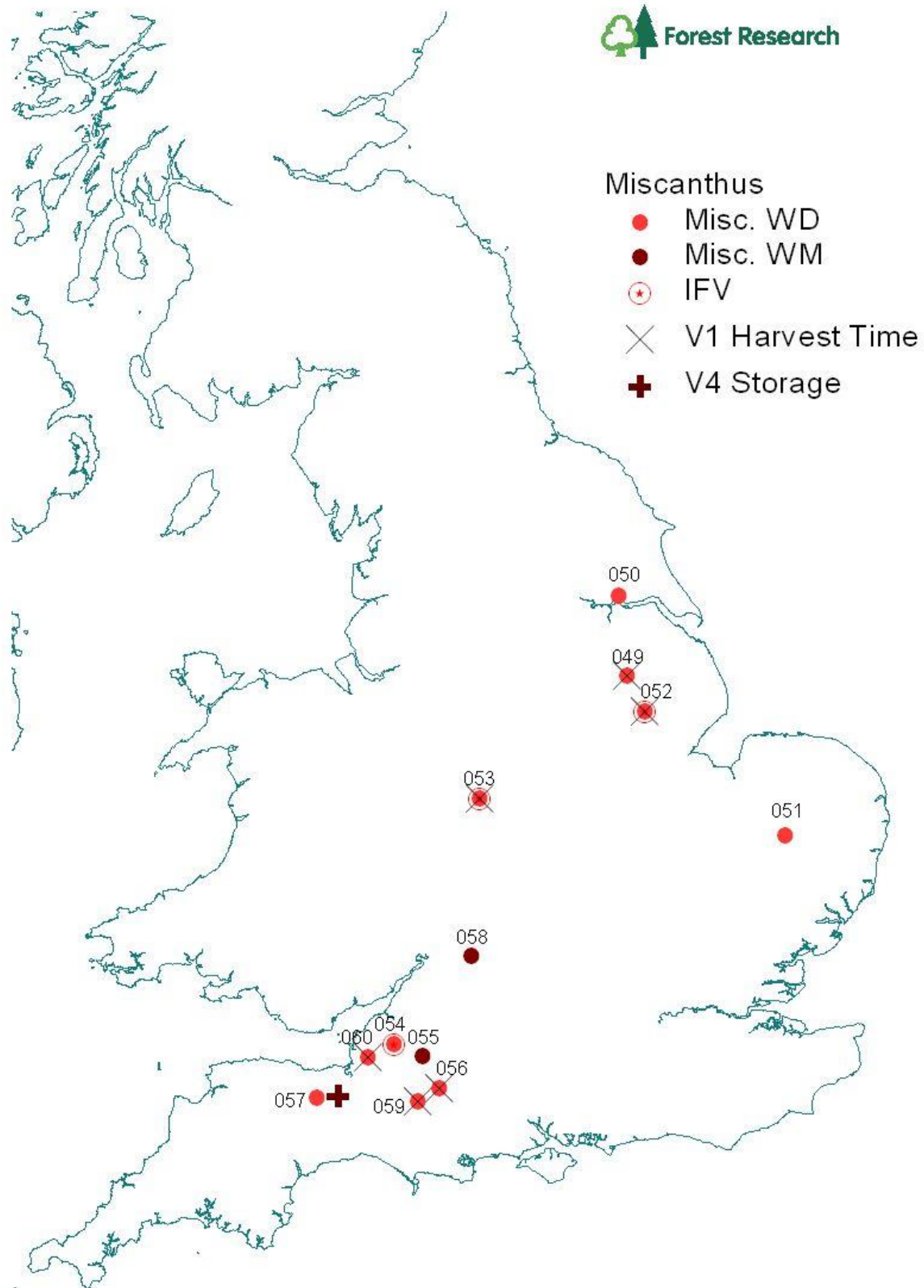
Table 2-1:   Overview of sampling undertaken in the four Investigations (n= the number of samples). Note that date format is day.month.year.

Feedstock	Climatic zone	Soil types	Harvest Time	Varieties	Time of Sample
Study 5: The impact of harvest time on the feedstock characteristics of *Miscanthus*					
Miscanthus	Warm/dry (n=6)	Light (n=3) Medium (n=3)	4 to 9.11.2015 4 to 12.01.2016 7 to16.03.2016 22.03 to 10.05.2016 27.04 to 26.05.2016	*Miscanthus* x giganteus	3 simulated harvests 1 sampling at commercial harvest 1 sampling pre-baling
Study 6: The impact of harvest time on the feedstock characteristics of willow SRC					
Willow SRC	Warm/dry (n=5) Warm/moist (n=1)	Light (n=5) Medium (n=1)	9 to 24.11.2015 8 to 25.01.2016 14 to 23.03.2016	Representative mix of commercial varieties	3 simulated harvests
Study 7: The impact of variety on the feedstock characteristics of willow SRC					
Willow SRC	Various (n=5)	Various (n=5)	29.02 to 3.03.2016	Endurance Tora Terra Nova Resolution Sven Nimrod	1 simulated harvest
Study 8: The impact of storage system and duration on the feedstock characteristics of *Miscanthus*					
Miscanthus	Warm/moist (n=1)	Medium (n=1)	18.4.2016	*Miscanthus* x giganteus	4 different storage systems – sampled monthly. May – November 2016

Feedstock characteristics in spring 2016 were compared with two different datasets collected in spring 2015. One possible comparison investigated was between the simulated harvests carried out between November 2015 and March 2016 and the simulated harvest in spring 2015 which investigated in-field variation. A second comparison was possible for the four sites which were sampled in spring 2015 and 2016 at both commercial harvesting and pre-baling (sites 49, 52, 56, and 59) which allowed a comparison of *Miscanthus* characteristics in the two years. Two further sites (Sites 53 and 60) had samples taken at commercial harvest in 2015 and 2016, but in 2015 the crop was baled almost immediately after cutting, with minimal time lying in the swath before baling, consequently it was pointless to take further samples. Since there was only one data point for each site and time, a statistical analysis was not possible, therefore insights into the feedstock condition in the two years are based on a visual interpretation of graphs.

2.2.2 Study 6: The impact of harvest time on the feedstock characteristics of willow SRC

Six willow SRC sites were selected, with two from the same holdings as used in Phase 1 where there were fields of harvesting age available for sampling in both 2015 and 2016 but four new sites had to be found to complete the set (see Figure 2-2). Since the objective was to examine the impact of harvest time on the inherent properties of the crop we looked for sites on the basis of predicted soil type to give in total three sites from each of two predicted soil types since at that point we expected soil type to be a significant influence on crop properties. In reality soil analysis confirmed that there was one medium soil type and five light soils. For the same reason, sites were sought from within only one climate zone in an attempt to limit the differences from one sampling time to the next due to local climate effects. Because of difficulties in finding sites within the warm dry climate zone, one site, which was in north-west England, was located in the warm moist climate zone.

On three occasions covering the autumn and winter (mid-November 2015, mid-January 2016 and mid-March 2016), samples were collected from standing crops at the normal cutting height to simulate commercial harvesting of the mix of varieties on the site. Summary details are provided in Table 2-1 and full details are included in the database D9. As described fully in D2, on each harvesting date 10 representative samples were collected from each site. The 10 samples were bulked, thoroughly mixed and sub-sampled to give a representative sample for each site and harvesting time.

Soil samples were collected at the first feedstock sampling occasion from all sites following protocols described in D2 for Phase 1.

Figure 2-2: **Map showing the location of willow SRC sites used in Study 6 (marked with a X) and Study 7 (marked with a *) in Phase 2 in relation to the sites used in Phase 1.**

Note that at Sites 13 and 16 the location of the field used is accurately located making the symbols appear slightly off centre. IFV = in-field variation; SRC-W- WD = Willow SRC Warm/dry climate zone; SRC-W- WM = Willow SRC Warm/moist climate zone; LEAF = leaf samples from Phase 1; V2 = Study 6; V3 = Study 7 from Phase 2.
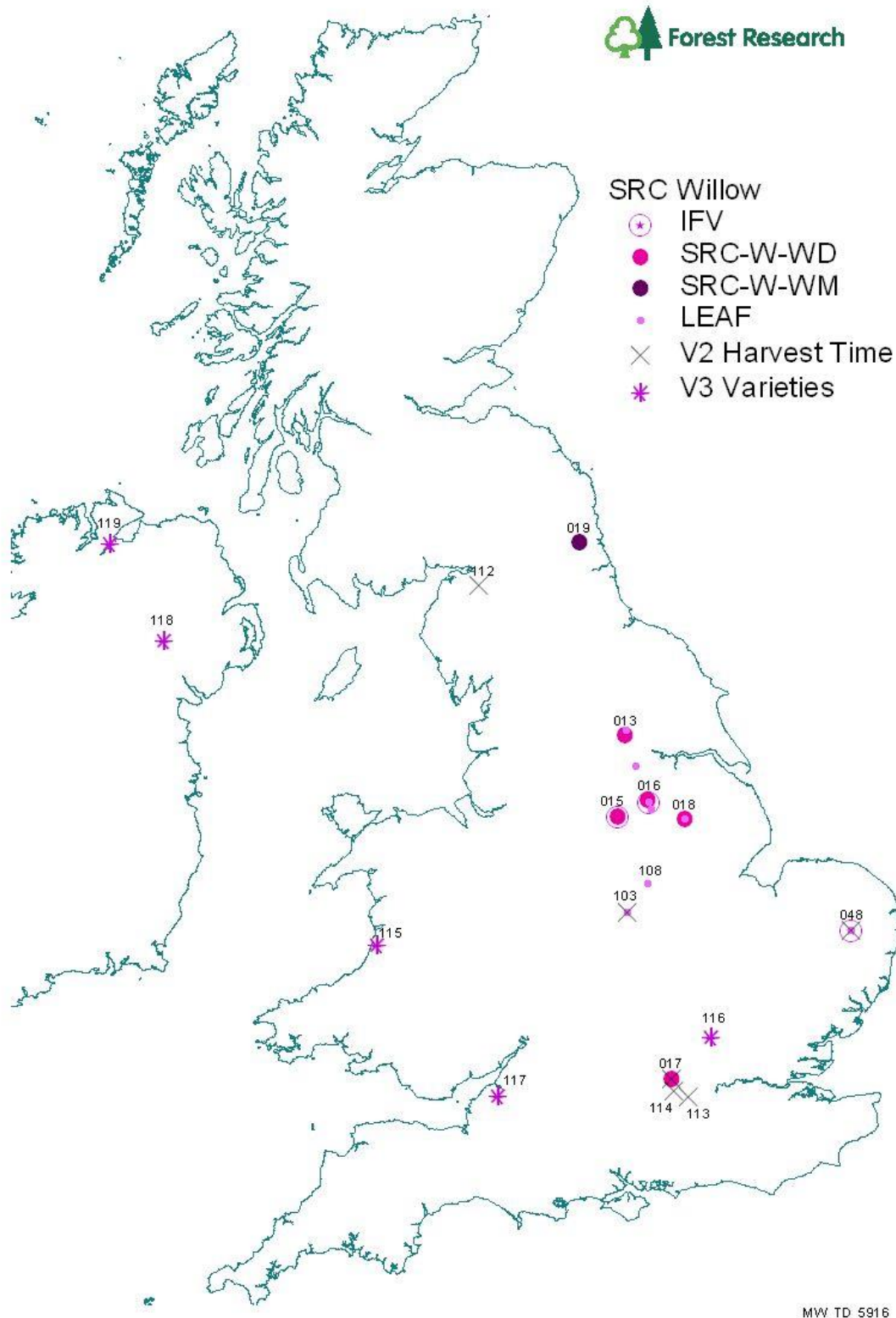
Two different sets of data are available from Phase 1 spring 2015 to compare with the samples collected from the simulated harvests collected in Phase 2 between November 2015 and March 2016. Firstly, a number of willow SRC samples were taken during commercial harvests in spring 2015. While all the varieties present on each site will have been included in these samples, the method of sampling is different to that used for the simulated harvests undertaken for this project variation and this may have affected some characteristics. Conversely, the second dataset from spring 2015 was collected by a simulated harvest, with multiple samples taken from three individual fields to investigate in-field variability. While these samples for in-field variation were taken using the same methods as in the simulated harvests of Phase 2 November 2015 - March 2016 sample collections, in the former case only one willow SRC variety (Tora) was sampled to minimise the number of variables. Note that although site 48 was used in both the 2015 in-field variation study and in Study 6, different fields were sampled, so year on year comparison should be made only with caution.

2.2.3 Study 7: The impact of variety on the feedstock characteristics of willow SRC

Five sites were selected (Aberystwyth, Rothamsted, Long Ashton, Loughall, Brook Hall; see Figure 2-2). These sites spanned a wide range of environmental conditions to provide a robust test of the consistency of willow SRC feedstock characteristics across a range of varieties. It was not possible to find commercial sites with a range of willow SRC varieties that could be definitely identified and located on the site, consequently the sites were generally trial sites run by research organisations.

Sampling at all sites was done within one week (end February/beginning of March 2016) to minimise the impact of sampling time on feedstock characteristics. As described fully in D2, on each harvesting date 10 waypoints were identified in advance and 5 to 10 (depending on weight of stem collected) representative samples were collected from each site to make up the required total sample weight. These samples were bulked, thoroughly mixed and sub-sampled to give a representative sample for each site and harvesting time.

Soil samples were collected at all sites on the same day as the biomass samples following protocols described in D2 of Phase 1.

In total six varieties (Endurance, Tora, Terra Nova, Resolution, Sven, and Nimrod) were identified that were found at all five sites with the exception of Sven and Nimrod which were absent at Aberystwyth. Newer varieties were available at some but not all sites, and since the main objective was to test the hypothesis that feedstock characteristics are consistent across sites we accepted older varieties that were found across a wider range of sites. Information about the lineage of the varieties is listed in Table 2-2 (Lindegaard, 2013). Summary details are provided in Table 2-2 and full details are included in the database D9.

Table 2-2: **Varieties of willow included in Study 7 and their parentage.**

Male	Female	Male	Female
Male		Female	
L78101 (S. viminalis)	L78195 (S. viminalis)	Unknown	Unknown
Orm (S. viminalis)		L79069 (S.schwerinii)	
Tora (Female)			
Orm (S. viminalis)	L79069 (S.schwerinii)	L830201 (S. viminalis)	N81102 (S. viminalis)
Björn (S. schwerinii x S. viminalis)		Jorunn (S. viminalis)	
Sven (Male)			
Unknown	Unknown	Unknown	Unknown
77056 (S.dasyclados)		(S. redheriana)	
Endurance (Female)			
Björn (S. schwerinii x S. viminalis)	Pavainen (S. viminalis)	Björn (S. schwerinii x S. viminalis)	Jorunn (S. viminalis)
Quest		SW930812	
Resolution (Female)			
Unknown	Unknown	Dark Newkind (*S. triandra*)	Bowles Hybrid (*S. viminalis*)
Shrubby willow (*S. miyabeana*)		LA940140	
Terra Nova (Female)			
Unknown	Unknown	Orm (S. viminalis)	L79069 (S.schwerinii)
Shrubby willow (*S. miyabeana*)		Tora (S. schwerinii x S. viminalis)	
Nimrod (Female)			

Blocks shaded in green are varieties from the Swedish Svalöf Weibull breeding programme; Blocks shaded in yellow are varieties from the UK Long Ashton breeding programme.

2.2.4 Study 8: The impact of storage type on *Miscanthus* fuel quality

It is generally considered that the correct storage method of *Miscanthus* is important in maintaining the fuel quality properties of the harvested and stored bales. Study 8 evaluated the possible effects of four different, but commonly used commercial storage systems, to understand what fuel quality improvements may occur during 6 months of storage and also what losses or degradation may occur during that time.

Study 8 evaluated four different storage methods for approximately 100 tonnes of *Miscanthus* bales; the choice of storage methods was based on the results of a questionnaire answered by 20 *Miscanthus* growers. The questionnaire, which collected information on for example storage methods and storage durations, was performed in conjunction with Terravesta and is provided in full in Appendix 8. Terravesta is a contracting company growing *Miscanthus* and canes and deals with several thousand hectares of *Miscanthus* production in the UK each year.

The majority (15) of the 20 surveyed growers stored their bales in either a fully enclosed barn or a partially enclosed shed (three sides and a roof), with three growers storing their bales outside under sheets, and only one grower storing the bales outside with no sheeting. In general, the choice of system was determined by what storage was available on their farm; only one grower had built dedicated storage. Nearly all growers used only one storage method each year and the same method was used year after year; only a few growers used a mixture of two methods each year, because of the number of bales they produce. At one location, bales were not stored because they left the farm straight away. Only one grower used external contractors to store their bales. Nearly all growers used a tele-handler to handle their bales, with many using a 2- or 3-bale grab, and a few using single bale spikes or tines.

Very few of the fully enclosed sheds had dedicated ventilation systems, with many growers stating it is not a necessary requirement for *Miscanthus* bales. No dedicated drying systems were currently being used and the general view was that sheds with open sides or gale-breakers (slated wooden boarding), which allowed airflow, were satisfactory.

The majority of bales stored inside (fully enclosed and partially enclosed) were stacked directly on the ground, with shed floors being typically concrete or consisting of loose stone, so not jeopardising the moisture content of the bales. Bales stored outside were also often stored directly on the ground, however sometimes bales were stored on a sheet, pallet, or something similar to keep the bales off the ground. No grower used any other type of straw bale as a buffer along the bottom or top of an outdoor stack, and no grower discarded the bottom layer, even when stacked directly on the ground.

When bales were stored outside, some stacks were not sheeted at all. Of the stacks that were sheeted, the top was always sheeted and although there was often an intention to cover the sides facing the prevailing winds, limits on sheet availability and ongoing maintenance requirements often made it less routine to sheet the sides. The best locations for outside storage were mainly determined by accessibility for lorries.

Survey responses indicated that the time bales remained on farm varied between growers and also year on year. Seven growers stored bales for less than 3 months, six growers stored bales for 3-6 months and seven used longer storage times. Terravesta commented that each year they alter the collection rota for their growers, in order to make storage delays fair for all growers. At certain locations however bales must be collected immediately, due to a high risk

of criminal damage, whilst other sites can easily hold their bales for >6 months and are happy to do so.

The four different storage methods evaluated (see Figure 2-3) were:

- Storage outside with no cover
- Storage outside with waterproof sheeting protecting the top of the stack of bales and the sides of the upper two/three bale layers only
- Storage inside in a building with open sides providing roof cover only
- Storage inside in a fully enclosed building

Figure 2-3:   Photographs of storage types tested

Top left – Storage outside with no cover; top right - Storage outside with waterproof sheeting protecting the top of the stack of bales and the sides of the upper two/three bale layers only; bottom left - Storage inside in a building with open sides providing roof cover only; bottom right - Storage inside in a fully enclosed building.

The full procedure used to store and sample bales in each of the four storage methods is provided in Appendix 9. A summary of this is as follows:

For each of the storage methods outlined above, the rectangular bales were stored in stacks of 48 bales; as: 5 bales high, 2 bales wide and 5 rows deep (except the top row which only had 4 rows – or 8 bales in the layer). The orientation of the rectangular bales can be seen in the top images in Figure 2-3.

In order to see if there were any noticeable differences between leaving the stack untouched (to replicate a typical commercial situation) in comparison to splitting and reassembling the stack each month which was necessary to ensure representative sampling from the bales' surface and interior faces, each of the four stacks was treated as two parts (i.e. the front 24 bales and the back 24 bales). The front part of each stack was dismantled each month for six months to allow representative sampling from the bales' surface and interior faces; by dismantling the stack the bales could also be sampled safely at ground level, see **Error! Not a valid bookmark self-reference.**. This is described later as repeated sampling. The back part of each stack was only sampled in May 2016 at the start (month 0) and at the final sample timing in November 2016; this is described later as start/end sampling.

Miscanthus bales are typically densely packed. To be representative of the biomass delivered to the end user, material had to be collected from the interior and not just the surface of the bales. To follow changes over time, it was decided to sample repeatedly from the same bales within the stack. Since the bales could not be split to gain access to the interior, a long-shafted corer was used (see next section for full description).

The bales of *Miscanthus* typically weighed 550 kg each and had a dimension of up to 2.5 m long, by 1.3 m wide and 1 m tall. The bales required moving by use of a dedicated bale grab, which was powered by a JCB telehandler, the grab can be seen in Figure 2-5.

2.2.5 Bale corer technology

The bale corer technology was sourced from Star Quality Biomass Samplers in Canada. The bale corer, seen pictured in Figure 2-6 was imported to the UK in spring 2016 and was the first time the device had ever been trialled on *Miscanthus* bales. The corer was powered by a hand-held rechargeable drill. At each sampling time, multiple cores were taken by drilling in from all but the underneath face of the bales sampled (i.e. from the top, sides and ends). A risk assessment was produced for ensuring the safe use of the bale corer. Cores were collected at monthly intervals from all bales located in the front part of each stack and one sub sample of approximately 1kg was then selected for final analysis; a similar sampling and subsampling procedure was followed on the back part of each stack at the beginning and end of the experiment.

The bale corer has a small diameter (1.3 cm) which made collection of the cores very slow. One complete sample per stack took up to 4 hours to collect; adding up to a total of two full days each month and four days for the first and last sample collection timings.

Figure 2-4: **Uncovered outside stored bale stack dismantled and bales ready for sampling. Outside covered bale stack can be seen in the back ground of image.**

Figure 2-5: **Image of the dedicated bale grab attachment.**

Figure 2-6: **Left – image of the corer bit; right – the subsample of *Miscanthus***

2.2.6 Storage site

The site selected for the trial was in Taunton, Somerset at the farm of a *Miscanthus* grower, Richard Gothard of *Miscanthus* Nurseries Ltd; the location is shown in Figure 2-1. Richard has been growing *Miscanthus* since 1999, and has a biomass boiler on site which uses his chipped *Miscanthus* to heat workers' accommodation, a B&B premises, and a site office at the farm. Richard also sells some of his annual harvest to Terravesta, so has a good understanding of typical storage requirements and timings required for commercial customers like Terravesta who supply Drax.

The *Miscanthus* bales used for the trial were harvested in the field next to the yard at the end of April 2016. The *Miscanthus* was left to dry in the field for almost three weeks prior to being baled and brought in to the farm yard and placed in to the four different storage systems. Typically Richard would store all his *Miscanthus* bales either in his fully enclosed barn or under his roof only storage system if for a shorter time.

2.3 Data analysis and evaluation

2.3.1 Overview

Analytical and provenance data from Investigations 1, 2, 3 and 4 have been added to the database generated in Phase 1 and submitted now as D11.

In view of the large dataset generated by the project, the project team developed a process to focus on the most important findings. This is described in detail in Section 2.6 and Section 3 of D6. In essence, the data were evaluated using statistical analysis in combination with the project team's understanding of the energy and heat sector and conversion technologies to focus on those parameters which are most influential. A flow chart describing the steps used to focus on the results to analyse and then illustrate in the project report are shown below (Figure 2-7).

The statistical approaches specific to Phase 2 are given below in Sections 2.3.3 while the analytical and operational evaluations are described in Sections 2.3.4 and 2.3.5 respectively. An abbreviated form of the process to select key variables relating to biomass feedstocks from the standard fuel analysis is given in Section 3 of this deliverable.

2.3.2 Quality assurance of data

Quality assurance of data was identical to the approach described in Section 2.3.2 of D6, which gave a very objective systematic assessment of all outliers. Many of the values identified as statistical outliers were retained as they were considered to be within the normal variation of the biomass feedstock. These are indicated by green shaded cells within the D11 database; those flagged values which were deemed to be "true" outliers are shaded red in D11.

Figure 2-7: Flow diagram to show process used to select data points to analyse and characteristics to highlight

RELEVANCE TO STUDIED FEEDSTOCKS Is the chemical analysis relevant to clean biomass feedstocks (as opposed to coal or waste wood)?	— No → Colour column heading in database red and do no further analysis on this parameter.

Yes ↓

EVALUATION OF STATISTICAL OUTLIERS Is the individual value <2 standard deviations of the mean?	— No → **EVALUATION OF FUNCTIONAL OUTLIERS** Is the individual value highlighted by the statistical evaluation within historical experience?

Yes ↓ (and Yes ← from functional outliers)

No ↓

IMPACT OF TIME OR VARIETY Is the probability of the main effect occurring by chance alone higher than 1 in 20, i.e. $p>0.05$	— No → Exclude individual value from further analysis but present as red cell in Database

Yes ↓

TABULATE RESULTS and proceed to ANALYTICAL EVALUATION (1) For each parameter, were most of the data above the limit of detection? [See Database]	— No → Exclude parameter from further analysis but present in Appendices

Yes ↓

ANALYTICAL EVALUATION (2) For each parameter, were the differences greater than the normal level of reproducibility?	— No → Exclude parameter from further analysis but present in Appendices

Yes ↓

OPERATIONAL EVALUATION For each parameter, are the differences between means likely to make an operational difference in the conversion plant?	— No → Exclude parameter from further analysis but present in Appendices

Yes ↓

PRESENTATION IN MAIN TEXT

2.3.3 Statistical analysis

Statistical analysis was used to evaluate the probability of a particular effect happening by chance alone. Effects were considered as not significant if the probability of it happening by chance alone was more common than 1 in 20, i.e. with $p>0.05$, whereas effects that were less common, i.e. with $p<0.05$, were considered worth further discussion.

Restricted Maximum Likelihood (REML) (Patterson and Thompson, 1971) models were fitted with site as a random effect. For Studies 5 and 2, time was fitted as a fixed effect and significance, where present, indicated a difference between mean response across sites at different time points. No time order structure is implied, e.g. decrease or increase with time.

For Study 7, Willow SRC variety was fitted as a fixed effect and significance, where present, indicated a difference between the varieties response, averaged across sites. Determination of variety by site interaction is not possible, so the general pattern of varietal differences should be considered indicative only.

A further assessment of the consistency of the varieties' ranking across sites was done using Kendall's Coefficient of Concordance which is a measure of association between K rankings on N individuals. For our data:

- The individuals are Willow SRC varieties with N=6
- The rankings are based on performance at K=5 sites
- Rankings are derived from a single measure of variety performance per site

The data are represented in a table with N=6 rows (variety) and K=5 columns (sites). The table cells represent the performance of a variety at a site. The varieties are then assigned a rank within each site and the rankings are compared across sites. Since two of the six varieties did not occur at one of the sites this analysis compared all six varieties across the four sites where they are fully represented.

All analyses in Investigations 1-3 were carried out using statistical software Genstat (VSN International, 2013).

For each variable in Study 8, a preliminary analysis assessed the significance of the difference between the initial and final mean value of each feedstock characteristic. To analyse the impact of storage type and duration, the difference was calculated for each data point versus the initial measurement in May; this removed any non-linear time responses and simplified the data for analysis, which was required due to the limited number of data points.

The first analysis compared differences between repeated sampling (monthly) and those samples taken only at the beginning and end of the experiment (6 months). Analysis of variance was conducted to assess whether these two treatment groups showed significant differences.

The second analysis differed for those variables where monthly data were recorded and for those where start/end data were recorded:

- For the monthly data, a range of linear mixed-effects models were applied to each variable to determine the effect of storage regime (factor) and time (covariate), using an information theoretic approach (Akaike Information Criteria, AIC). This approach considers both the fit of the model and the number of parameters, and generates an AIC that reflects both (i.e. considers model efficiency). A mixed-effects model approach was used to account for the repeat measures design of the experiment (i.e. that the

same single replicate was measured each month). This was accounted for in the model by incorporating replicate as a random effect (for the intercept and/or time covariate). The range of models applied to the data included 1) an interaction between storage regime and time, 2) an additive effect between storage regime and time only, 3) a single effect of storage regime, 4) a single effect of time, and 5) a fixed intercept only. The AIC was calculated and compared for each of the five models, and the model with the lowest AIC selected in each case, as this represents the model with the best fit given the number of parameters included within the model (most efficient model).

- For the start/end data, a simple analysis of variance was applied to the November data (change in variable since May) to determine whether the four storage regimes showed significant differences. Where significant differences existed, post-hoc tests were conducted to determine where the differences lay between the four regimes (pairwise comparison of the least-square means for each treatment (e.g. 1 vs. 2, 1 vs. 3, etc.) with Bonferroni adjustments to the p value (0.05 adjusted for 6 comparisons).

The following software packages for the statistical system R were used:

- Base R package (R Core Team, 2016) – pre-loaded package for basic statistical functions
- Package "car" (Fox & Weisberg, 2011) – ANOVA (start/end data)
- Package "lme4" (Bates *et al.*, 2015) – Mixed-effects models (monthly data)
- Package "lsmeans" (Lenth, 2015) – least-square means (start/end data)
- Package "multcompView" (Graves *et al.*, 2016) - least-square means lettering (start/end data)

These packages are add-ons, required for specific statistical analyses, available to download within the R programme via the CRAN website (https://cran.r-project.org/web/packages/available_packages_by_name.html).

In all analyses, appropriate assumptions of the models were assessed (normal distribution, homoscedasticity of residuals etc.).

2.3.4   Analytical evaluation

Two criteria were used:

1. the analytical limit of detection – impacts were not interpreted further if the majority of the data were at the limit of detection (LOD), on the grounds that any variation between these data are misleading because any values that were below the LOD were assumed to be present at the LOD, even though they were probably lower.
2. the analytical error reproducibility – impacts were not interpreted further if the means of the statistically significant effects were closer than the normal level of reproducibility achieved by different accredited labs when subsamples of the same original material are analysed (as defined by the relevant standards).

2.3.5   Operational evaluation

Impacts were not interpreted further if the differences between means of the statistically significant effects would make no operational difference, usually because the values were all well below important thresholds.

3 Key parameters and justification

3.1 Fuel parameters

For all conversion technologies, proper matching of the fuel and equipment is important. Failure to understand the probable impacts of the feedstock on the system is likely to result in reduced efficiency, lower availability, increased OPEX and increased emissions. Different conversion technologies will have different acceptable levels for each feedstock parameter. These limits will depend on a number of factors, such as steam parameters and technology type and will tend to be more restrictive for those technologies offering the highest quality outputs (e.g. highest efficiency or specific conversion products).

The most common fuel analyses undertaken can be divided into six main categories; proximate analysis, ultimate analysis, trace element content, ash composition, ash fusion temperatures and physical properties. For the purpose of this study, the analysis options were divided into the groups as follows:

A   Proximate and ultimate analyses (moisture, ash, volatile matter, net calorific value, gross calorific value, sulphur, chlorine, carbon, hydrogen, nitrogen)

B   Ash composition (SiO_2, Al_2O_3, Fe_2O_3, TiO_2, $CaCO_3$, MgO, Na_2O, K_2O, Mn_3O_4, P_2O_5, BaO) plus trace metals (Ba, Be, Cr, Co, Cu, Mo, Ni, V, Zn)

C   Extended trace metals (Hg, Pb, Cd, As, Se, Sb)

D   Halides (bromine and fluorine)

E   Ash fusion temperatures.

It is industry practice to express the majority of species on a dry fuel basis, allowing easy comparison between feedstocks, but other bases are also used; in particular the proximate analysis is often compared on an "as received" (wet fuel) basis for moisture and net calorific value (NCV) and dry, ash-free basis for gross calorific value (GCV) and volatile matter (VM). Note that in most of the figures the data are presented on a dry basis (mg/kg or % weight in the fuel), however for the statistical analysis the major fuel elements (C, H, N, S, Cl) were expressed on a dry, ash-free basis to enable any fundamental changes in chemical composition to be examined without being impacted by changes in ash.

A number of these analyses are of limited interest for many conversion technologies, particularly for clean feedstocks, but are analysed in conjunction with other components.

Some of the trace elements in particular were consistently found at levels below (or close to) the limit of detection (LOD) in some or all of the feedstocks in this project, restricting detailed review. Of those components which do warrant in-depth investigation, some are key fuel quality parameters, others may affect boiler performance (for example through impacts on slagging and fouling, corrosion and bed agglomeration) while some are of environmental concern. Using these criteria, prioritisation of the analysed parameters for statistical review was determined (see Table 3-1). The critical levels of these fuel components will vary depending on the conversion system and, for those of environmental concern, installed clean-up equipment.

3.2 **Soil and site parameters**

As well as the feedstock samples, a (composite) soil sample was collected for each site in Studies 6 and 3 and submitted for analysis (data for all *Miscanthus* sites in Study 5 were already available from Phase 1). The analyses were identical to those in Phase 1 as described in Section 3-2 of D6. Phase 1 had indicated that there were almost no significant relationships between feedstock characteristics and the soil properties at the typical rural sites which were low in contaminants. Nevertheless soil samples were also collected in Phase 2 to confirm soil type and classification and to allow further exploration of any unexpected values. The rationale for the selection of soil properties relevant to Phase 2 is shown in Table 3-2. Results of all soil analysis can be found in the D11 database.

3.2 **Soil and site parameters**

Table 3-1:   Analysed fuel parameters and their prioritisation for statistical review

Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
A - Proximate and Ultimate analysis	As Received fuel basis	Moisture content wt %	High	High moisture content will reduce combustion plant efficiency. Potential impact on fuel handling/ dustiness/ degradation in storage
		Fixed carbon wt %	Low	Calculated from other parameters – limited value
		NCV kJ/kg	High	Direct impact on size and efficiency of plant and fuel logistics
	Dry Fuel Basis	Ash wt %*	High	Impacts calorific value, plant efficiency, slagging and fouling tendencies, erosion. Ash handling systems need to be designed to deal with the expected ash quantities of ash produced. With high ash levels, fluidised bed materials may need more regular replacement.
	Dry, Ash-free basis	Volatile matter wt %	High	Volatile matter will impact on flame stability, combustion burnout performance and NOx emissions.
		GCV kJ/kg	High	Measure of fuel consistency across different feedstock samples - allows comparison without being affected by moisture and ash
		Carbon wt %*	Medium	Measure of fuel consistency across different feedstock samples - limited direct impact on plant although will affect CO_2 emissions per unit output; slight impact on CV
		Hydrogen wt %*	Medium	Measure of fuel consistency across different feedstock samples – used in NCV calculations
		Nitrogen wt %*	High	Direct impact on NO_x emissions

Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
		Sulphur wt %*	High	Direct impact on SO$_x$ emissions and can be corrosive in high temperature systems, but in lower temperature systems can mitigate against chloride corrosion. Acid gases also cause amine degradation in carbon capture processes.
		Chlorine wt %*	High	Implicated in corrosion mechanisms and acid gas emissions. Acid gases also cause amine degradation in carbon capture processes.
		Oxygen wt %* (by difference)	Low	Calculated from other parameters – limited value; impact on CV
Category B - Trace elements	mg/kg dry fuel	Barium	Medium	Limited environmental/plant impact
		Beryllium	High	Emissions are of environmental concern
		Chromium	High	Emissions are of environmental concern
		Cobalt	Medium	Limited environmental/plant impact
		Copper	High	Emissions are of environmental concern
		Molybdenum	Medium	Limited environmental/plant impact
		Nickel	High	Emissions are of environmental concern
		Vanadium	Medium	Limited environmental/plant impact
		Zinc	High	Emissions are of environmental concern. Implicated in corrosion mechanisms. Metallic zinc can melt in combustion systems and block air nozzles (not expected to be an issue for clean feedstocks)

Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
Category C - Trace elements	mg/kg dry fuel	Antimony	High	Emissions are of environmental concern
		Arsenic	High	Emissions are of environmental concern. Poison for NO_x reduction catalysts.
		Mercury	High	Emissions are of environmental concern.
		Selenium	High	Emissions are of environmental concern
		Cadmium	High	Emissions are of environmental concern
		Lead	High	Emissions are of environmental concern. Can have an impact on plant integrity. Elevated levels in the ash and boiler deposits may also be of occupational health concern to plant workers
Category D - Halides	mg/kg dry fuel	Bromine	High	Forms acidic gases which are of environmental concern and may be involved in corrosion mechanisms. Also believed to damage bag-house filters but may aid in mercury capture mechanisms. Acid gases also cause amine degradation in carbon capture processes.
		Fluorine	High	Forms acidic gases which are of environmental concern may be involved in corrosion mechanisms. Acid gases also cause amine degradation in carbon capture processes.

Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
Category B - Ash forming elements in fuel	mg/kg dry fuel - back-calculated from measured concentration of the oxide in ash	Aluminium	High	Alumino-silicate in the ash may mitigate alkali-metal mediated corrosion/ slagging/fouling
		Calcium	High	Principal biomass ash component - impacts on slagging. May help acid gas abatement
		Iron	High	High levels of iron in ash can cause slagging, but it is normally present at low concentrations in biomass
		Potassium	High	Key concern for plant corrosion and slagging. Alkali metals may also result in formation of fine particulate matter which is an issue for emissions and for amine-based carbon capture processes. Poison for NO_x reduction catalysts.
		Magnesium	High	Magnesium in the ash may mitigate alkali-metal mediated corrosion/ slagging/fouling
		Manganese	Medium	Manganese levels are normally so low in biomass ash as to have no significance
		Sodium	High	Key concern for plant corrosion and slagging. Alkali metals may also result in formation of fine particulate matter which is an issue for emissions and for amine-based carbon capture processes.
		Phosphorous	High	Poison for NO_x reduction catalyst. Phosphorous may also be implicated in corrosion.
		Silicon	High	Alumino-silicate in the ash may mitigate alkali-metal mediated corrosion/ slagging/fouling. Silica (quartz) may cause abrasion and erosion.
		Titanium	Medium	Titanium levels are normally so low in biomass ash as to have no significance

Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
Category E - Ash fusion temperatures	Reducing Atmosphere, °C	Initial deformation	Low	Data unsuitable for statistical analysis. Ash fusion temperatures provide an indication of the likelihood of ash slagging and bed agglomeration
		Softening	Low	
		Hemisphere	Low	
		Flow	Low	
	Oxidising atmosphere, °C	Initial deformation	Low	
		Softening	Low	
		Hemisphere	Low	
		Flow	Low	
Category B - Ash Oxides	Normalised ash oxides, %wt in ash (calculated from measured ash oxides to normalise for SO_3 and express Ca as $CaCO_3$)	Al_2O_3*	High	Alumino-silicate in the ash may mitigate alkali-metal mediated corrosion/ slagging/fouling.
		BaO*	Medium	Barium levels are normally so low in biomass ash as to have no significance
		$CaCO_3$*	High	Calcium is often the most dominant macroelement in biomass ash and can be implicated in slagging and fouling. May help acid gas abatement
		Fe_2O_3*	High	High levels of iron in ash can cause slagging, but it is normally at low concentrations in biomass
		K_2O*	High	Key concern for plant corrosion and slagging. Alkali metals may also result in formation of fine particulate matter which is an issue for emissions and for amine-based carbon capture processes. Poison for NO_x reduction catalysts.

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Analysis Group	Parameter basis	Parameter	Priority for statistical analysis	Justification/impact on conversion systems
		MgO*	High	Magnesium in the ash may mitigate alkali-metal mediated corrosion/slagging/fouling
		Mn_3O_4*	Medium	Manganese levels are normally so low in biomass ash as to have no significance
		Na_2O*	High	Key concern for plant corrosion and slagging. Alkali metals may also result in formation of fine particulate matter which is an issue for emissions and for amine-based carbon capture processes.
		P_2O_5*	High	Poison for NO_x reduction catalysts. Phosphorous may also be implicated in corrosion.
		SiO_2	High	Principal ash component - Alumino-silicate in the ash may mitigate alkali-metal mediated corrosion/slagging/fouling. Silica (quartz) may cause abrasion and erosion.
		TiO_2*	Medium	Titanium levels are normally so low in biomass ash as to have no significance
Derived	Alkali Index	$kg(Na_2O + K_2O)/GJ$	High	Measure of slagging risk in combustion systems

*Also included in D4 on other bases but these are considered to be lower priority for statistical review as they are less comparable

Table 3-2.   Analysed soil parameters and their prioritisation for statistical analysis

Provenance Data Collected	Reasoning for provenance data collection
Clay, Silt, Sand and Organic Matter (OM) % in soil	Understanding the clay, silt, sand and organic matter (OM) content within a soil is important in helping to understand the potential capacity for achieving element exchange between the plant and the soil.
Soil Classification – including higher classification of (Light, Medium and Heavy)	Soil classification is based on the ratios of clay, silt, sand and organic matter within a soil. The soil classification chart used is shown in D6, which identifies eleven textural classes and three organic classes. The textural classes are grouped further into Light, Medium and Heavy soils based on their mineral and/or organic soil classifications.
pH	The soil pH is required to understand any potential limitations or lock up of certain elements in the soil.
Elements analysed	The elements analysed for the project were the most common for plant health status, and those elements which are linked to both desirable and undesirable elements seen within biomass composition when being combusted. The main soil elements were analysed were; P, K, Mg, Ca, Na, Fe, Zn, Mn, Cl, S, N, plus Cu, B, Mo, Co, Se, Pb, As, Ni, Cd, Hg, Cr. All elements that were present above the Limit Of Detection (LOD) have been statistically analysed for correlations between feedstock characteristics and soil properties.
CEC (Cation Exchange Capacity)	The CEC of a soil helps to determine a soil's capability to hold and offer elements to a plant. The higher a CEC of a soil the more readily elements are able to be transferred within the soil and between the soil and the plant.

4 Results and interpretation

4.1 Study 5: The impact of harvest time on feedstock characteristics of Miscanthus

4.1.1 Changes during November 2015 to late May 2016

Many of the *Miscanthus* feedstock characteristics changed significantly through time (

Table 4-1) with the majority of the characteristics decreasing in value. Of these, many showed a decreasing trend throughout the period during the time both as a standing crop (November 2015 to March 2016) and also as the crop was harvested commercially and left on the stubble until baling. There were however interesting variants of this general decreasing trend. A smaller number of feedstock characteristics increased between November 2015 and June 2016 and in one case (see below) levels apparently increased through the winter and early spring but then decreased again. A standard simplified set of graphs of all feedstock characteristics at the five sampling times is included in Appendix 2.

A selection of the more interesting statistically significant seasonal patterns is given below. Examples of decreasing trend throughout the sampling period include moisture content where the average reduced from ~65% in November to 40% in March and then 17% at baling (see Figure 4-1), as well as carbon and chlorine (see Figure 4-2). For others e.g. ash (Figure 4-3), calcium (Figure 4-4), and silicon, the decrease was most pronounced at the start and slowed over time with little change between the March sampling, the commercial harvest and the pre-baling sample. Some interpretation of the results is included here while the results are discussed further in Section 5.

Figure 4-1: **Changes in moisture content of *Miscanthus* during November 2015 to late May 2016 by site**
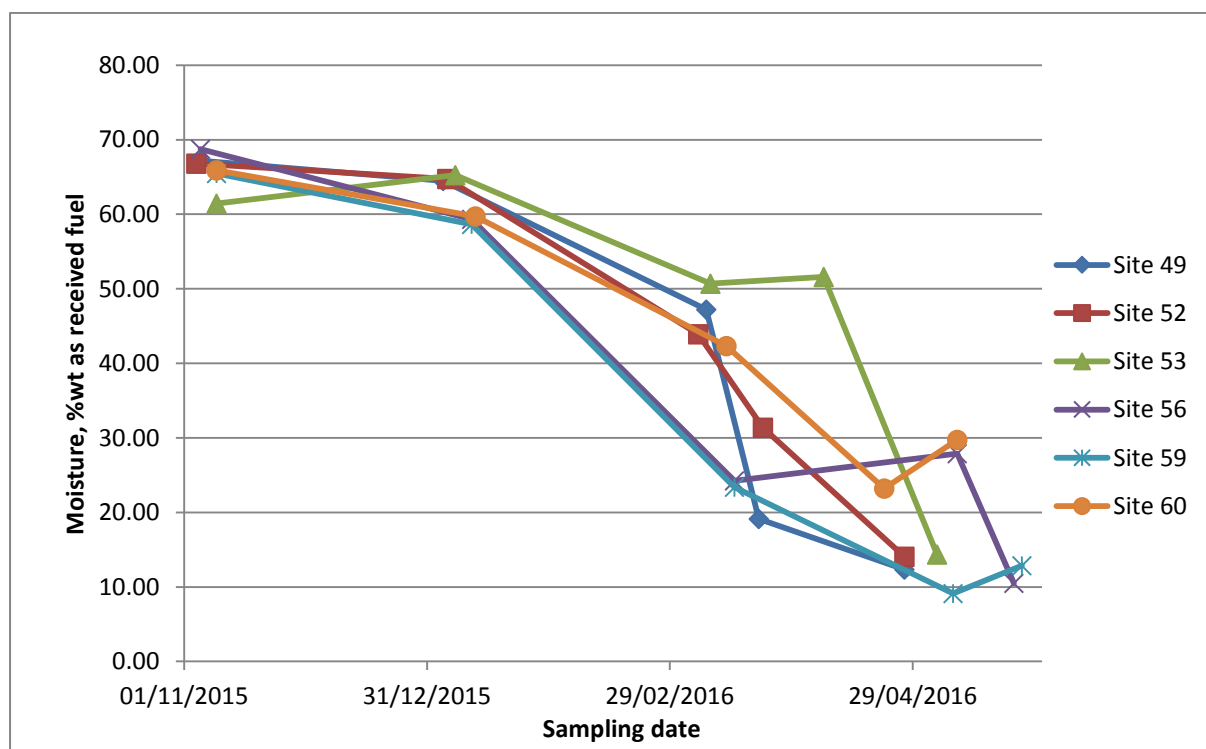
Table 4-1: Description of trends in *Miscanthus* characteristics during November 2015 to late May 2016

Variable (basis of analysis)†	Description of trends
Moisture (ar)	Decreased
Net calorific value (ar)	Increased
Ash content (d)	Decreased then stable
Volatile matter (DAF)	Increased
Gross calorific value (DAF)	Decreased
Carbon (DAF)	Decreased
Hydrogen (DAF)	x
Nitrogen (DAF)	Decreased then stable
Sulphur (DAF)	Decreased then stable
Chlorine (DAF)	Decreased
Barium (d)	Decreased
Beryllium (d)	N/A
Chromium (d)	x
Cobalt (d)	N/A
Copper (d)	x
Molybdenum (d)	Decreased then stable
Nickel (d)	N/A
Vanadium (d)	N/A
Zinc (d)	Decreased
Antimony (d)	N/A
Arsenic (d)	N/A
Mercury (d)	N/A but seemed to increase after cutting
Fluorine (d)	N/A
Bromine (d)	Decreased, main decrease November to January
Selenium (d)	N/A
Cadmium (d)	x
Lead (d)	x
Aluminium (d)	x
Calcium (d)	Decreased
Iron (d)	x
Potassium (d)	x
Magnesium (d)	x
Manganese (d)	x
Sodium (d)	Convex (decreasing then increasing)
Phosphorous (d)	Decreased
Silicon (d)	Decreased
Titanium (d)	Decreased over winter but often increased at baling
Al_2O_3 (na)	x
BaO (na)	x
$CaCO_3$ (na)	Decreased then stable
Fe_2O_3 (na)	x
K_2O (na)	Increased then stable
MgO (na)	x
Mn_3O_4 (na)	x
Na_2O (na)	Increased over winter then erratic
P_2O_5 (na)	x
SiO_2 (na)	x
TiO_2 (na)	Convex (decreasing then increasing) over winter then slight increase
Alkali index	X

†basis of analysis; ar = as received fuel, d = dry fuel, DAF = dry, ash-free fuel, na= normalised ash.
x = no statistical impact of time, N/A = values below or close to limit of detection therefore trends are not interpreted

Figure 4-2: Changes in chlorine content of *Miscanthus* during November 2015 to late May 2016 by site
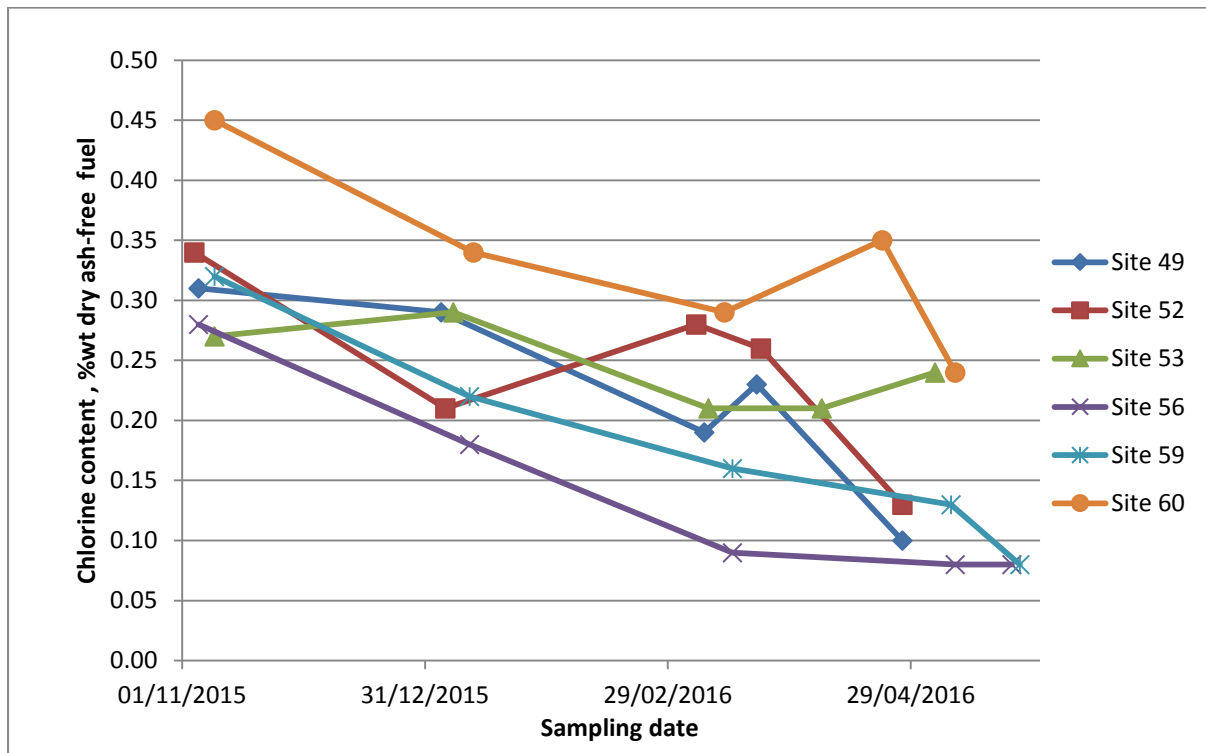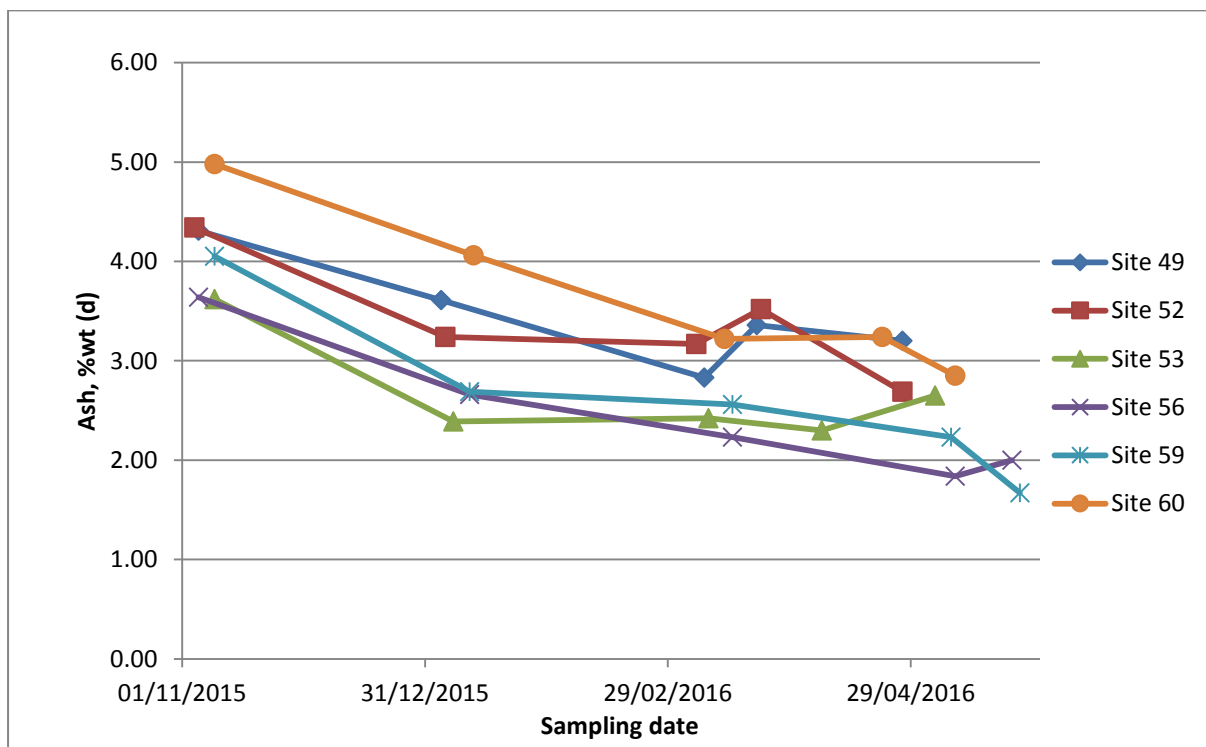

Figure 4-3: Changes in ash content of *Miscanthus* during November 2015 to late May 2016 by site


Broadly speaking, changes that are associated more closely with passive processes, e.g. moisture content, fell in a more linear way throughout the period, for example at sites 60 and

59 the concentrations of chlorine can both be seen to drop in line with moisture content. Trends showing a decrease during the winter and early spring, e.g. ash contents, are consistent with a perennial crop where active growth has ceased; there is no longer uptake of water, nutrients, trace elements or metals; and in addition there is active translocation of energy and nutrients to the rootstock to support growth of the new shoots the following spring. As the moisture levels drop the plant starts to sequester carbon to the rhizomes. Chlorine levels will fall in line with leaf material dropping from the plant and a natural leaching effect due to rainfall through the autumn and winter. Loss of leaf material and natural leaching may also explain the significant drop in sulphur and bromine.

We anticipated that concentrations of components that are regulated by the plant would decline most noticeably in the autumn and change little in the spring when the *Miscanthus* stem is essentially inert. This general pattern seemed to hold for ash and calcium, however this was also the pattern shown by silicon which is not mobile. The leaves of *Miscanthus* have very high levels of silicon; as these drop to the ground during the winter senescence the silicon level in the standing crop can be seen to reduce. It should also be considered that harvest operations (post commercial cutting) could cause silicon levels to increase via contamination from soil or rain splash events.

Figure 4-4: **Changes in calcium content of *Miscanthus* during November 2015 to late May 2016 by site.**

N.B. the high value recorded for Site 56 in early March was flagged as a statistical outlier but was not rejected because the value was within historical experience
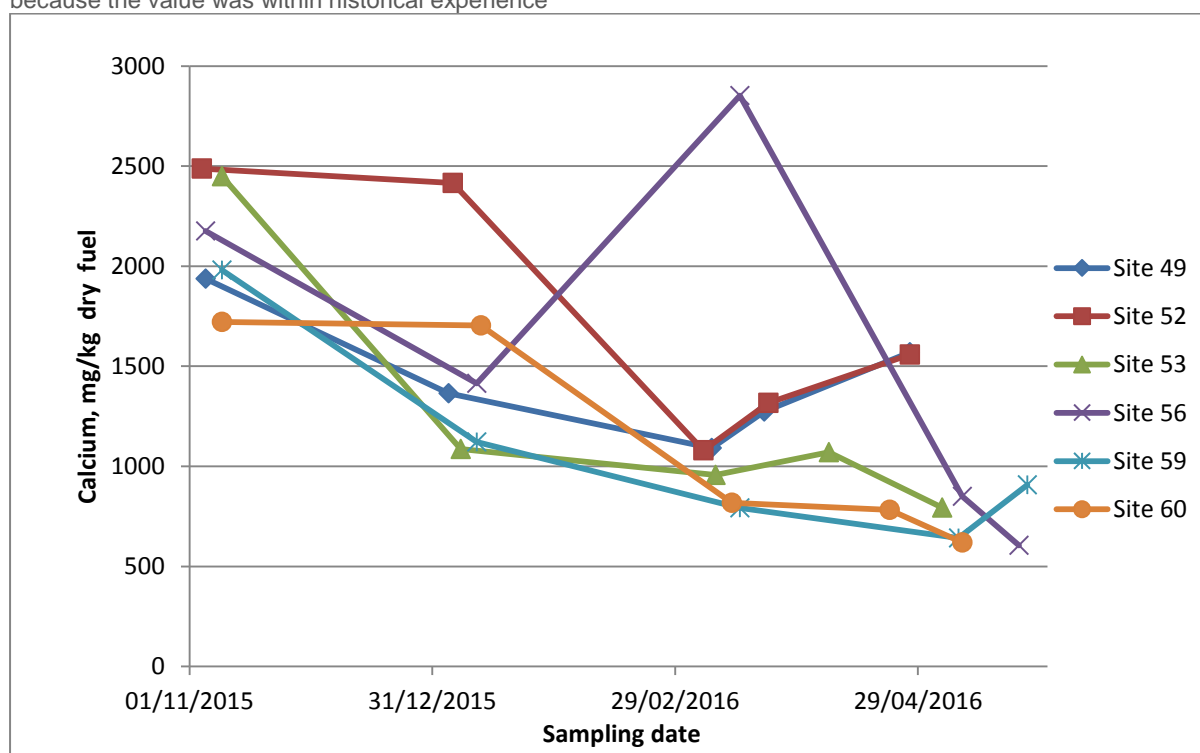

Within the general trend of declining values over time, there was a third group which showed a slight increase in the values in spring, especially between the March simulated harvest and the actual commercial harvest, for example nitrogen (Figure 4-5), gross calorific value, molybdenum, and titanium. In the case of nitrogen there is a view among *Miscanthus* growers in the UK (Steve Croxton, pers. comm.) that winters in some more southerly areas are not reliably cold enough to always complete the annual growth cycle of the stems with the result

that, when conditions improve in the spring, nutrients and sugars are remobilised and translocated to the overwintered stem to support continuation of previous season's growth, before starting the new season's growth. In sub-tropical and tropical environments *Miscanthus* does not typically senesce over the winter period as growth is continual, so this re-growing in the spring after a mild autumn/winter is typical behaviour for the species in warmer climates.

In the case of the increase in metals in the late spring, it is more likely that the increase reflects contamination during commercial harvesting or the period when the harvested stems were lying on top of the stubble before baling.

Figure 4-5: Changes in nitrogen content of *Miscanthus* during November 2015 to late May 2016 by site
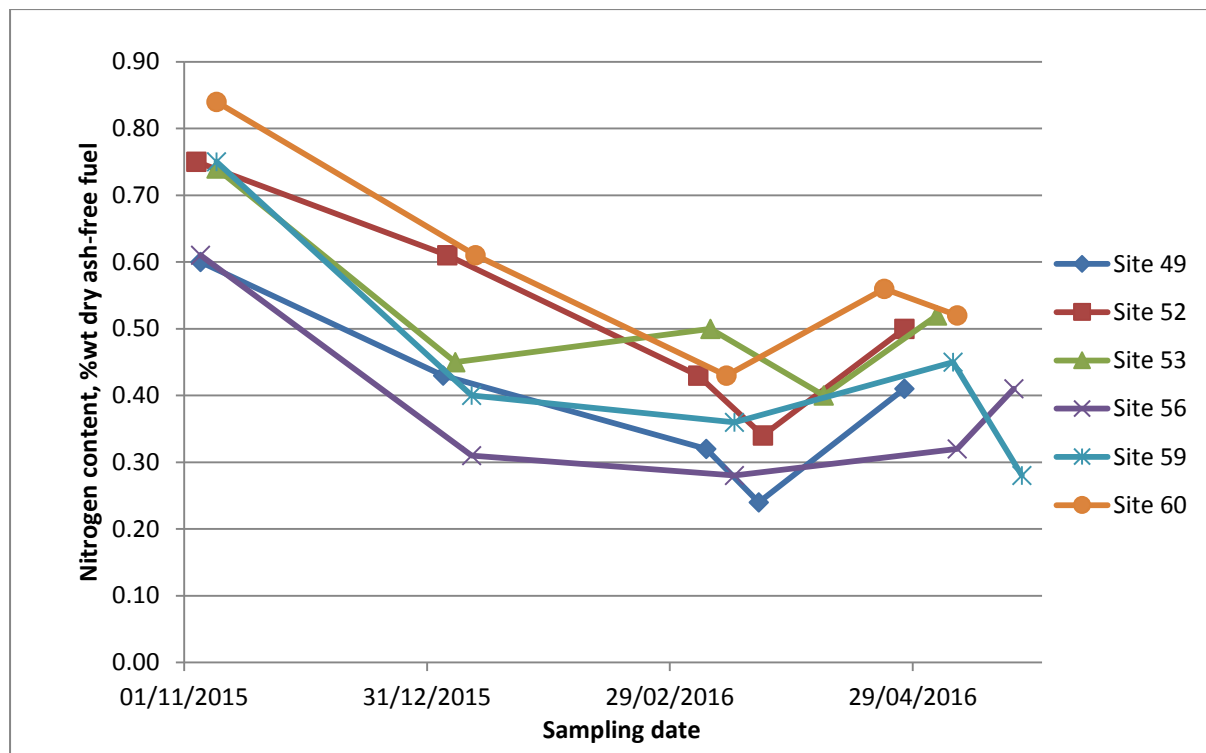

Two characteristics, net calorific value and volatile matter, increased throughout the sampling period (Figure 4-6). Since net calorific value increases as moisture content falls, it is not surprising that Figures 4-1 and 4-6 show opposite trends.

Figure 4-6: Changes in net calorific value of *Miscanthus* during November 2015 to late May 2016 by site
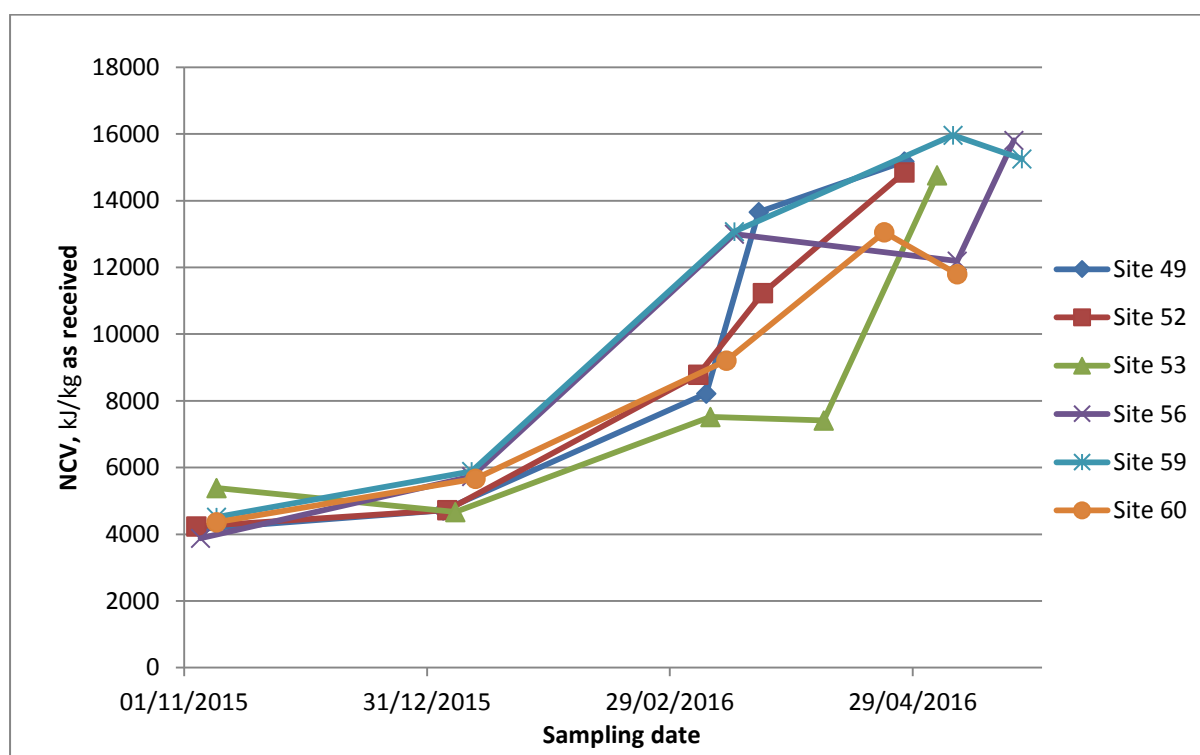
Sodium had a unique hump-backed pattern during the sampling period, which was remarkably consistent across the six sites (Figure 4-7). In general the sodium levels increased between November and January and then continued to increase but at a slower rate until March after which they tended to level off (although concentrations at some sites continued to increase and some decrease). Despite the range of response between the March simulated harvest and the commercial harvest, *Miscanthus* from almost all sites had lower levels by the time of the pre-baling sampling compared to the levels observed at commercial harvesting. A possible explanation for the increase is due to remobilisation of salts within the plant prior to spring new growth and the decrease around actual harvest timing is probably due to leaching and loss of external leaves and stem sheaths.

Figure 4-7: **Changes in sodium levels of *Miscanthus* during November 2015 to late May 2016 by site**
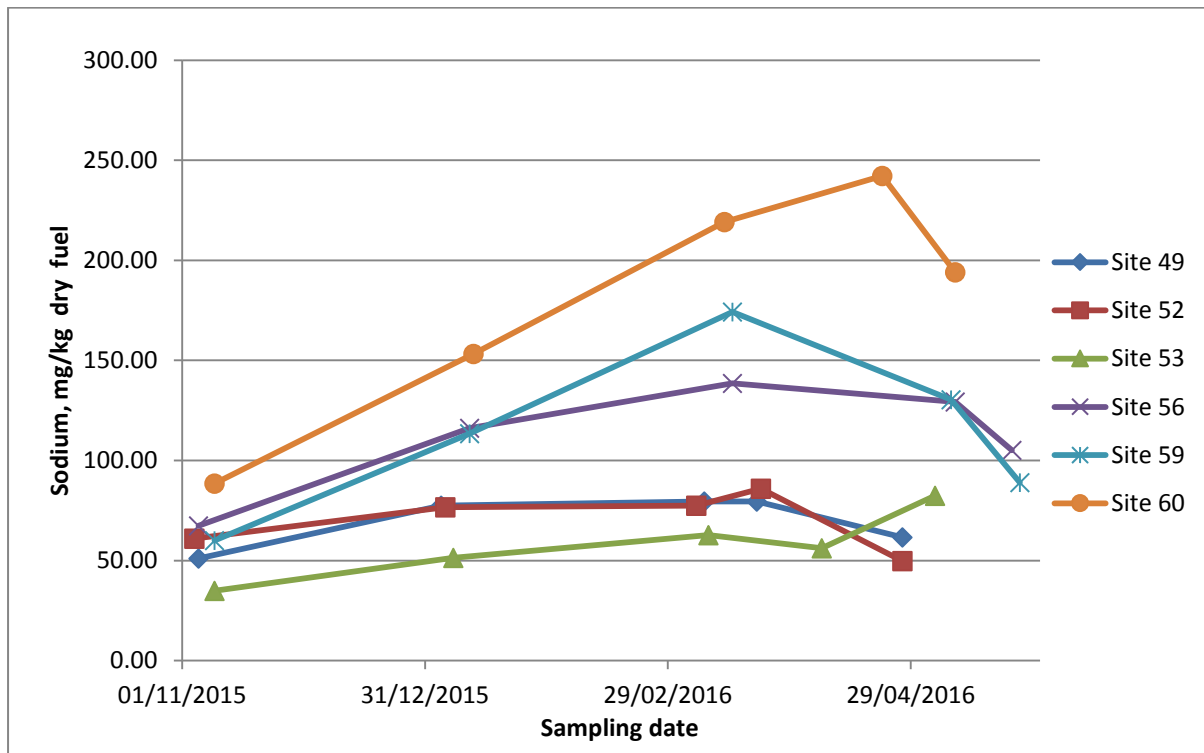

4.1.2 Comparison of spring 2015 and spring 2016

To recap on Section 2.2.1, four sites had samples taken in both spring 2015 and 2016 at commercial harvesting and pre-baling (sites 49, 52, 56, and 59). Two more sites (Sites 53 and 60) had samples taken at commercial harvest in 2015 and 2016 – unfortunately the crop was baled within a very few days of cutting so there was no justification for taking a pre-baling sample immediately after the harvest sample.

The following observations focus on the characteristics that showed clear seasonal patterns between November 2015 and June 2016 (see Appendix 3). A selection of the more interesting statistically significant seasonal patterns is given below. It is immediately clear from Figure 4-8 (compare dashed lines and open symbols which represent 2015 with solid lines and symbols of the same colour which represent 2016 at the same site) that the moisture content fell at much the same rate between commercial harvesting and pre-baling at the four sites where a comparison was possible. Comparing the simulated harvests of the In-field variation study in Phase 1 (red bar symbols) with the simulated and actual harvests of Phase 2, the two sequences are well aligned through March. Moreover, for a given calendar date and site, the crop was drier in 2015 during this stage in the crop handling; the two samples collected at harvesting in Sites 53 and 60 were also drier in 2015 (compare the single green triangles and orange dot, which represent 2015 with the lines of the same colour and symbols which represent 2016).

Figure 4-8:   Changes in moisture content in spring 2015 and 2016.

Solid lines and symbols indicate 2016 results, dashed lines/hollow symbols indicate 2015 data from sites with the equivalent colour and symbol shape.
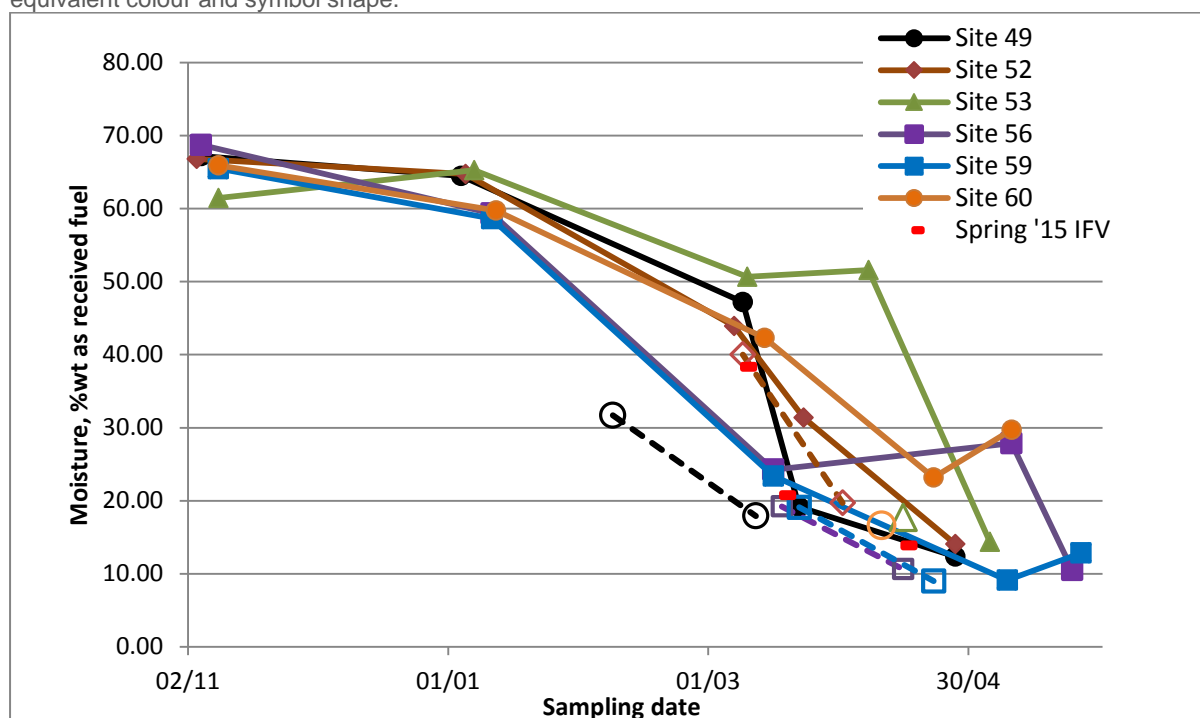

The differences in net calorific value between the two years follow the observed differences in moisture content as expected. Consequently NCV levels were higher site for site in 2015.

Ash content of the *Miscanthus* taken from crops in the two years showed a range of differences (Figure 4-9). In terms of ash levels, three sites were very close in the two years (Sites 49, 53 and 56), two sites had lower levels in 2015 (Site 52 and 60) while one had a higher level of ash in 2015 (Site 59). The trends in ash content for both years were broadly similar *for the time of year* with a decrease at three sites and an increase at one site. However *comparing the same harvesting stage*, i.e. harvesting through pre-baling, the behaviour in ash content was less consistent across the two years. The average of the ash content of 2-2.5 % from the simulated harvests for the in-field variation study of Phase 1 were towards the lower end of that seen in samples from the simulated harvests of the six sites the following year.

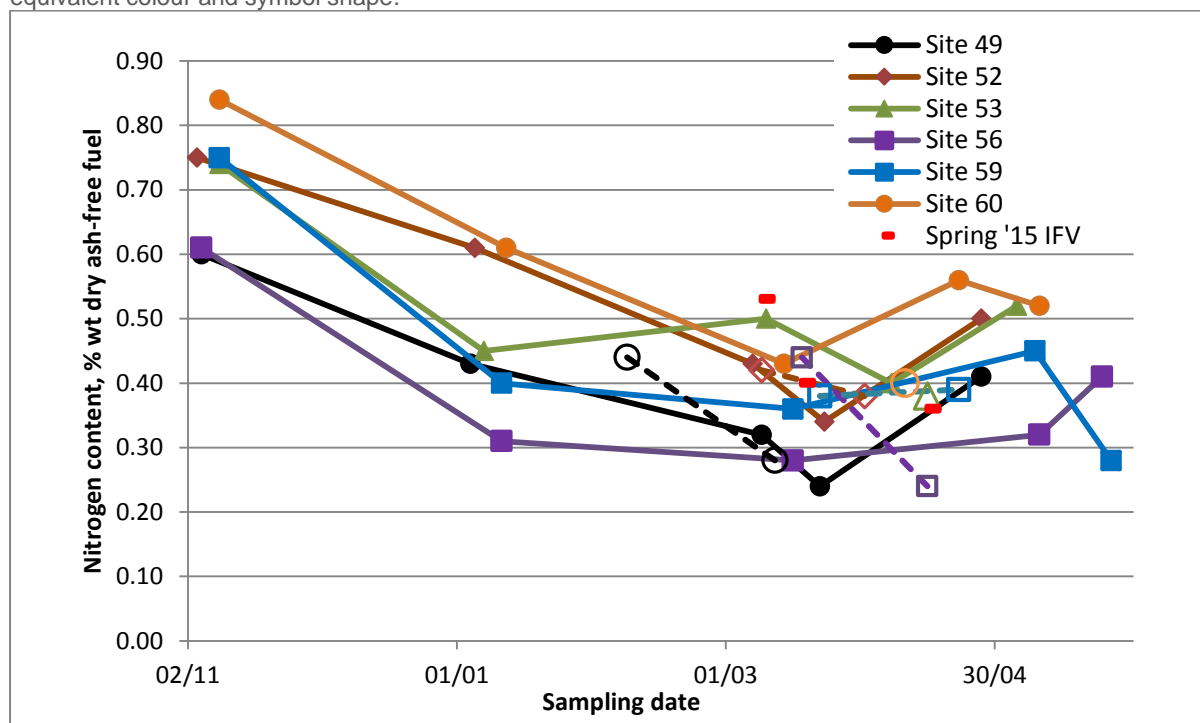**Figure 4-9: Changes in ash levels in spring 2015 and 2016.**

Solid lines and symbols indicate 2016 results, dashed lines/hollow symbols indicate 2015 data from sites with the equivalent colour and symbol shape.
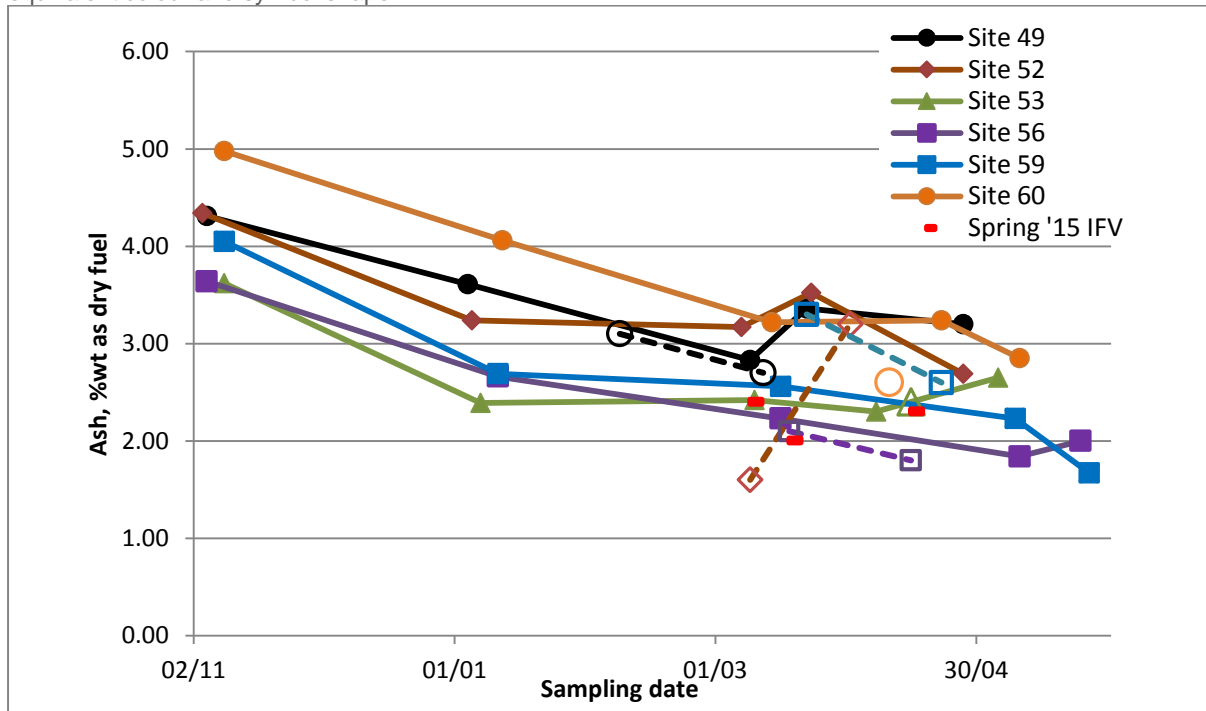
Nitrogen levels in the *Miscanthus* had a broad range, with differences possibly linked to both site and time of year (Figure 4-10). Nitrogen concentrations were broadly similar in the two years under investigation, with a range of 0.24 to 0.55 % wt dry ash-free fuel. Unlike 2016 when N levels between harvesting and pre-baling increased at four sites, the N levels in spring 2015 declined or remained the same during the equivalent period.

**Figure 4-10: Changes in nitrogen levels in spring 2015 and 2016.**

Solid lines and symbols indicate 2016 results, dashed lines/hollow symbols indicate 2015 data from sites with the equivalent colour and symbol shape.

When the same calendar dates are compared in 2015 and 2016, chlorine levels were generally lower in 2015. When the same harvesting stages are compared, chlorine levels were approximately the same in both years (Figure 4-11). The changes in chlorine content of the *Miscanthus* during the harvesting stage in 2015, which were slight declines at three of the four sites, were less apparent than in 2016 when three sites showed quite marked declines in chlorine between harvesting and pre-baling.

Figure 4-11:   Changes in chlorine levels in spring 2015 and 2016.

Solid lines and symbols indicate 2016 results, dashed lines/hollow symbols indicate 2015 data from sites with the equivalent colour and symbol shape.
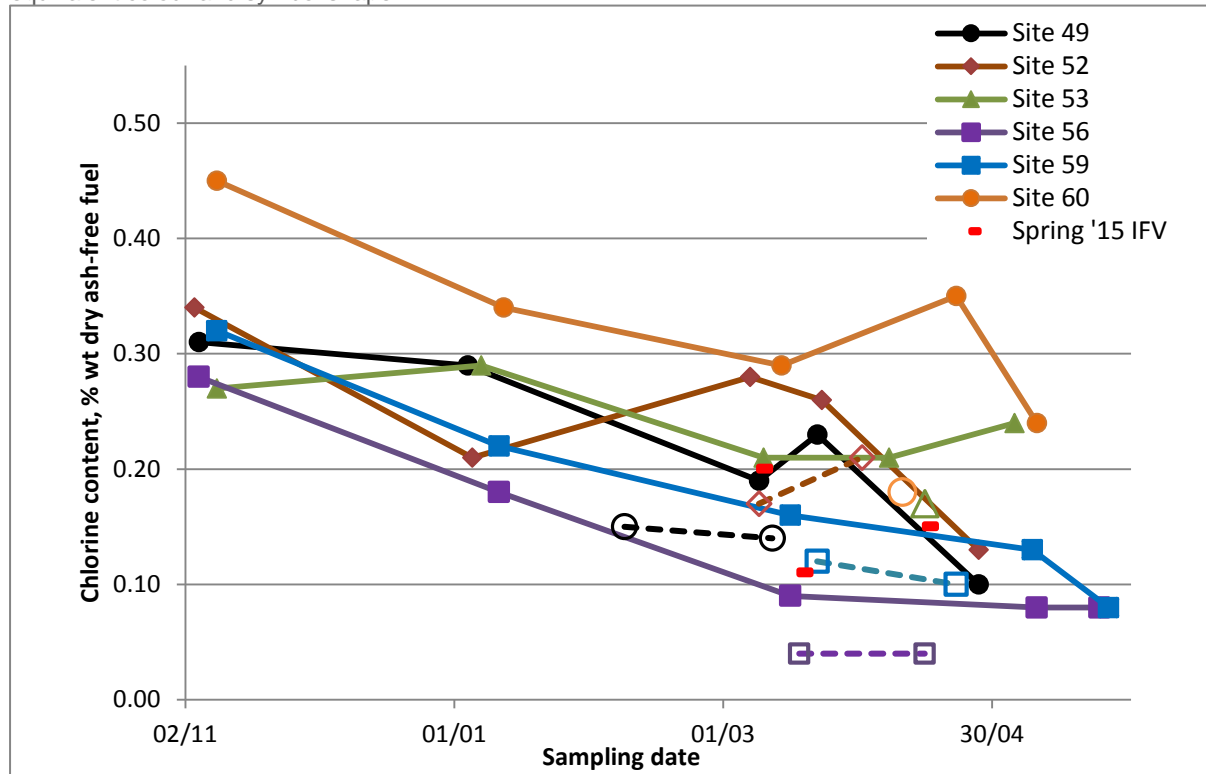

When the same calendar dates are compared in 2015 and 2016, calcium levels in the *Miscanthus* samples taken in 2015 were slightly higher than those in 2016. When the same harvesting stages are compared, calcium levels were approximately the same in both years between harvesting and pre-baling. There did not seem to be a consistent trend in levels during this period.

In 2015, sodium levels in *Miscanthus* taken from three sites were broadly similar to those observed in 2016 and in both years sodium levels declined between harvesting and pre-baling (see Figure 4-12). The fourth site however had a dramatically higher level in the pre-baling sample than at harvesting.

Figure 4-12: Changes in sodium levels in spring 2015 and 2016.

Solid lines and symbols indicate 2016 results, dashed lines/hollow symbols indicate 2015 data from sites with the equivalent colour and symbol shape.
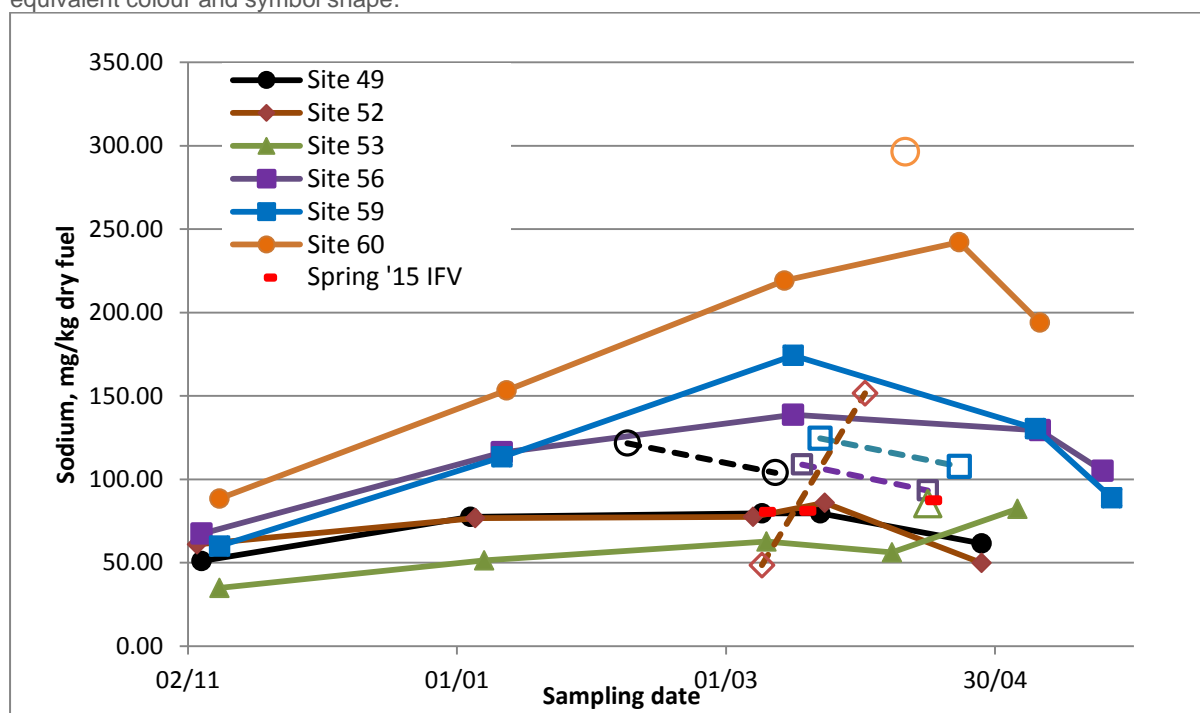
In summary, considering the results of both years, it was observed that many *Miscanthus* characteristics changed significantly through time and in the majority of cases the levels decreased through late autumn, winter and early spring, while the crop was still standing and after the crop was harvested and left on the stubble prior to baling. Over the same time, a much smaller number of characteristics rose in concentration. A comparison of *Miscanthus* characteristics at the same calendar dates in 2015 and 2016 suggested that date for date, the levels in the assessed characteristics were generally similar in the two years up to the point of harvesting. The trends were also similar in the two years. Changes between harvesting and the pre-baling sample, however, were less consistent across sites and years.

4.1.3   The effect of rainfall on *Miscanthus* characteristics

It was observed that the values of a number of parameters changed between the samples taken immediately after the *Miscanthus* crop was cut as part of the conventional harvest, and those taken immediately before the crop was collected for baling. This period is conventionally used to reduce the moisture content further, and it has been suggested it can also allow the loss of mineral content as a result of leaching. However it was observed that values of individual parameters changed differently over this period at different sites, including in a different direction, and also differently at the same site over the two different years for which we have data.

Met data were obtained to investigate the impact on feedstock characteristics of firstly rainfall between harvesting and baling in 2016 and secondly of rainfall during January to April in 2015 as compared with 2016.

One hypothesis was that differences in local rainfall during the time the crop was lying in the field led to different levels of washout of individual minerals. This was a potential effect considered during Phase 1 of the project and consequently rain gauges were placed in the

fields to measure local rainfall, however of the six Phase 1 sites of relevance to Phase 2 Study 5, two had been cut and baled immediately, leaving four sites with measured rainfall during the interval between cutting and baling which could be compared to 2016 data. Rain gauges were not installed in Phase 2, so daily rainfall figures were obtained from the nearest Meteorological Office measuring stations.

One further hypothesis is that it is not the total rainfall, but the intensity of rainfall that could lead to enhanced washout, consequently as well as total rainfall, average rainfall per day was also considered.

In Table 4-2 a number of parameters have been colour coded as to whether they increased or decreased over the time between cutting and baling. The depth of colour has been used to distinguish between those that changed slightly, or more significantly. Similarly depth of colour has been used to highlight levels of both total rainfall and average rainfall per day.

Table 4-2: **Rainfall between cutting and baling for _Miscanthus_ harvests during 2015 and 2016**

FR site No.	Days in the field	Total rainfall (mm)	Average rainfall (mm day^{-1})	Moisture content*	Cl	Ash	Ca	N	Na	K
2015										
049	31	21	0.68	DD	D	DD	U	DD	D	DDD
052	22	24	1.09	DDD	UU	UUU	UUU	D	UUU	UUU
053	0									
056	27	37	1.37	DD	L	D	D	DD	D	DD
059	29	35	1.21	DD	D	DD	DD	U	D	DD
060	0									
2016										
049	36	88	2.44	DD	DD	D	UU	UU	D	UU
052	35	68.8	1.97	DD	DD	DD	UU	UU	D	UUU
053	28	70.2	2.51	DDD	U	UU	DD	UU	UU	U
056	14	43.4	3.10	DDD	L	U	DD	UU	DD	UU
059	17	34.8	2.05	UU	DD	DD	UU	DDD	DDD	DD
060	17	26.4	1.55	UU	DDD	DD	DD	DD	DDD	UUU

Key to Table 4-2: * D=Slightly down; DD=Down; DDD=Steeply down; U=Slightly up; UU=Up; UUU=Steeply up; L=Level

It has not been possible to identify any unequivocal, simplistic correlations between either rainfall totals or intensity. Looking at moisture content (MC), two samples in 2016 increased in MC while in the field, however these included the two lowest total rainfall sites; in terms of average daily rainfall, these sites had the lowest and third lowest inputs. Although there were two periods of significant rainfall immediately before the 2016 pre-baling samples were collected from site 60, which showed increased MC, site 59 (the other site showing increased MC) had six days of no rainfall prior to the collection of the pre-baling samples.

In 2016, there appears to be a correlation between those sites in which nitrogen increased and higher total rainfall levels, however this was not evident in the 2015 results. It appears possible from the 2016 data that the highest average daily rainfall correlated with sites in which chlorine and ash levels increased, but this was not evident in the 2015 data.

It was therefore not possible to draw any firm conclusions, based on the current level of analysis, about the impact of rainfall on changes in parameters between cutting and baling the *Miscanthus* crop. We have looked at total rainfall and average rainfall per day, whereas it is possible that the timing and distribution of rainfall events, such as the peak rainfall intensity, or heavy rain immediately after cutting, might display greater correlation. Owing to the small number of data points during the period between harvesting and baling, it is felt there is limited potential for more detailed analysis.

Our purpose was to investigate the hypothesis that differences in local rainfall during the time the *Miscanthus* crop was lying in the field led to different levels of washout of individual minerals. The conclusion drawn was that if rainfall was a contributory factor, the relationship was not a straightforward correlation between change in parameters and either total rainfall or average daily rainfall.

4.1.4 Summary

- Many of the *Miscanthus* characteristics of crops grown at six sites ranging from Lincoln to the south west England changed significantly through time.
- In *Miscanthus* a general decrease through late autumn, winter and early spring was observed in moisture content, ash, carbon, nitrogen, chlorine, molybdenum, zinc, bromine, phosphorus, silicon, and calcium accompanied by an increase over the same period in net calorific value, volatile matter, and sodium.
- Considered as a whole these results suggest that to maximise *Miscanthus* quality, harvesting should be delayed until at least the beginning of March, with chlorine and ash a particular concern if harvesting is brought forward which also risks losing the advantages of low moisture content and higher NCV.
- Several sites, mainly those in south west England, showed a previously unreported pattern of increasing nitrogen in the late spring 2016 which may be associated with a resumption of growth in stems following mild winter conditions.
- It was not possible to draw any firm conclusions about the impact of rainfall on changes in parameters between cutting and baling the *Miscanthus* crop.

4.2 Study 6: The impact of harvest time on feedstock characteristics of willow SRC

4.2.1 Changes during November 2015 to March 2016

Only a few feedstock characteristics of willow SRC showed statistically significant differences across the three simulated harvesting times, with the majority showing no difference (see Table 4-3). Of the proximate and ultimate fuel properties only gross calorific value showed a significant change. For the characteristics that did change, a variety of patterns was evident.

Table 4-3: Description of trends in willow SRC characteristics through time

| Variable (basis of analysis)†	Description of trends
Moisture (ar)	x
Net calorific value (ar)	x
Ash content (d)	x
Volatile matter (DAF)	x
Gross calorific value (DAF)	Decreasing then increasing
Carbon (DAF)	x
Hydrogen (DAF)	x
Nitrogen (DAF)	x
Sulphur (DAF)	x
Chlorine (DAF)	x
Barium (d)	x
Beryllium (d)	N/A
Chromium (d)	Increasing
Cobalt (d)	x
Copper (d)	x
Molybdenum (d)	N/A
Nickel (d)	x
Vanadium (d)	N/A
Zinc (d)	x
Antimony (d)	x
Arsenic (d)	x
Mercury (d)	x
Fluorine (d)	x
Bromine (d)	x
Selenium (d)	x
Cadmium (d)	x
Lead (d)	x
Aluminium (d)	x
Calcium (d)	x
Iron (d)	x
Potassium (d)	x
Magnesium (d)	x
Manganese (d)	x
Sodium (d)	x
Phosphorous (d)	x
Silicon (d)	x
Titanium (d)	x
Al_2O_3 (na)	x
BaO (na)	x
$CaCO_3$ (na)	Stable then increasing
Fe_2O_3 (na)	x
K_2O (na)	Decreasing
MgO (na)	x
Mn_3O_4 (na)	x
Na_2O (na)	x
P_2O_5 (na)	Stable then decreasing

SiO$_2$ (na)	x
TiO$_2$ (na)	x
Alkali index	x

†basis of analysis; ar = as received fuel; d = dry fuel; DAF = dry; ash-free fuel; na= normalised ash; x = no statistical impact of time; N/A = values below or close to limit of detection therefore trends are not interpreted.

A standard simplified set of graphs of all willow SRC feedstock characteristics for samples taken at the three sampling times is included in Appendix 4. A selection of the more interesting statistically significant seasonal patterns is given below. Some interpretation of the results is included here while the results are discussed further in Section 5.

Gross calorific value of the willow SRC at all sites fell in samples taken between mid-November and mid-January, but by mid-March the GCV of samples was either similar or had increased again (see Figure 4-13). We attribute the fall between mid-November and mid-January to movement of various carbohydrates from the shoot to the root system for overwinter storage and the increase in spring to the remobilisation and movement back to the shoot (in response to environmental cues) to support early leaf growth.

Figure 4-13:   Changes in gross calorific value of willow SRC during November 2015 to March 2016 by site
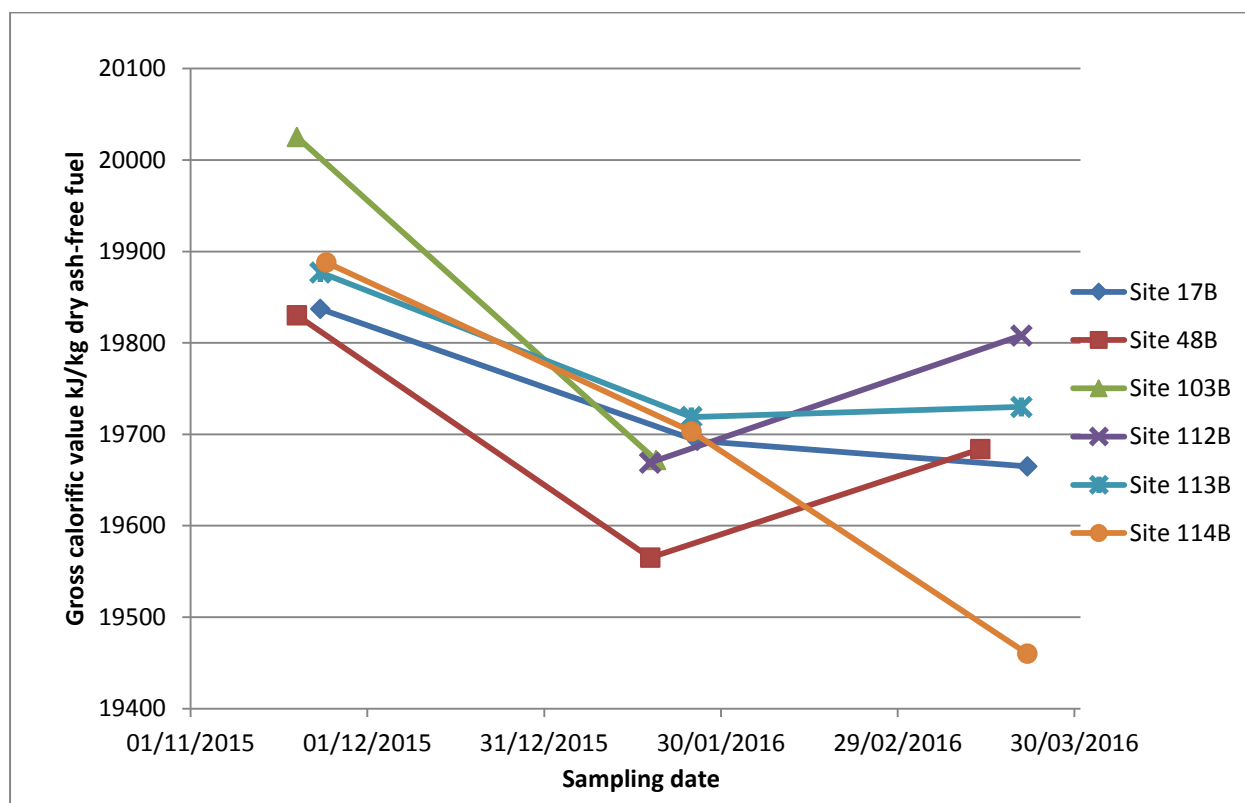
Generally, levels of chromium in the willow SCR increased with time (see Figure 4-14).

Willows are well known to be bio-accumulators, especially of metals, and the pattern observed in this study indicates that chromium continued to be taken up from the soil and accumulated in the shoots even when the willows were leafless.

Figure 4-14: Changes in chromium of willow SRC during November 2015 to March 2016 by site
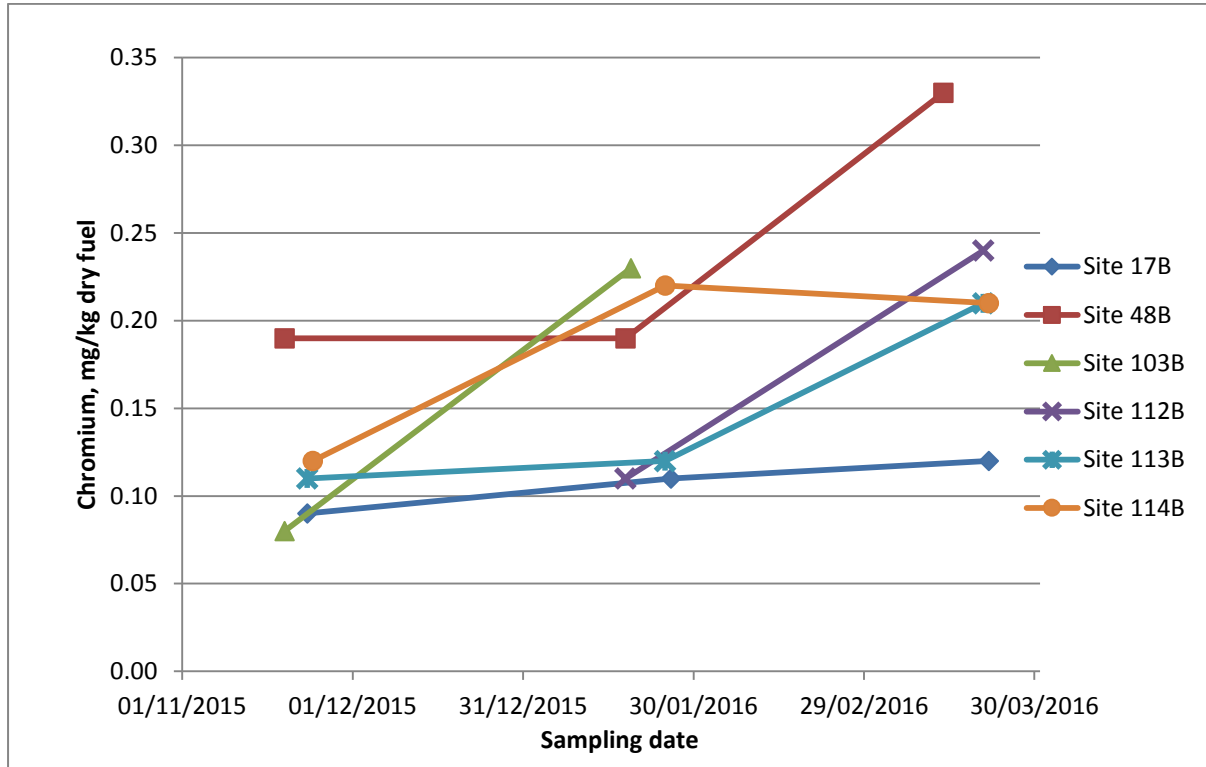
4.2.2   Comparison of spring 2015 and spring 2016

Willow SRC feedstock characteristics in spring 2016 can be compared with two different datasets collected in spring 2015. One possible comparison is the simulated harvests carried out between November 2015 and March 2016 with the simulated harvest in spring 2015 to investigate in-field variation. It should be noted that only one variety of willow was sampled in the study of in-field variation to minimise potential complications, whereas the simulated harvests in spring 2016 sampled the mix of varieties present at the site. One farm location (Site 48) was sampled in both years although different fields were sampled each year. A further comparison is the simulated harvests carried out between November 2015 and March 2016 with the samples collected in spring 2015 immediately after commercial harvesting. In both years the mix of varieties present on the site were sampled but the commercial harvesting process may have caused some contamination of the samples.

We considered only the characteristics showing changes over time in Phase 2 (see Table 4-3) and plotted the data points obtained in 2015 over the 2016 data points to compare the values at the same calendar date in the year (see Appendix 5). Of these, the most interesting was the comparison of gross calorific value (see Figure 4-15) which suggests that firstly the GCV was slightly higher in samples taken in 2015 and secondly that the observed increase continued throughout April and May. By the time of sampling in April and May 2015, the majority of sites had started to flush – in some cases early leaves were present and included

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in the sample taken for analysis. This is probably one of the main causes for increases seen in GCV during April and May 2015. The other trends suggested in Table 4-3 were not corroborated when spring 2015 sampling data were overlaid on spring 2016 data with the possible exception of P_2O_5, which as shown in Figure 4-16 seemed to decline slightly in samples between mid-January and in mid-March 2016 and also between mid-February and early May 2015. It is also interesting that Site 48, at which two different fields were sampled, had much lower levels of P_2O_5 than all the other sites.

Figure 4-15: Changes in gross calorific value in willow SRC in spring 2016 plus values in red from 2015
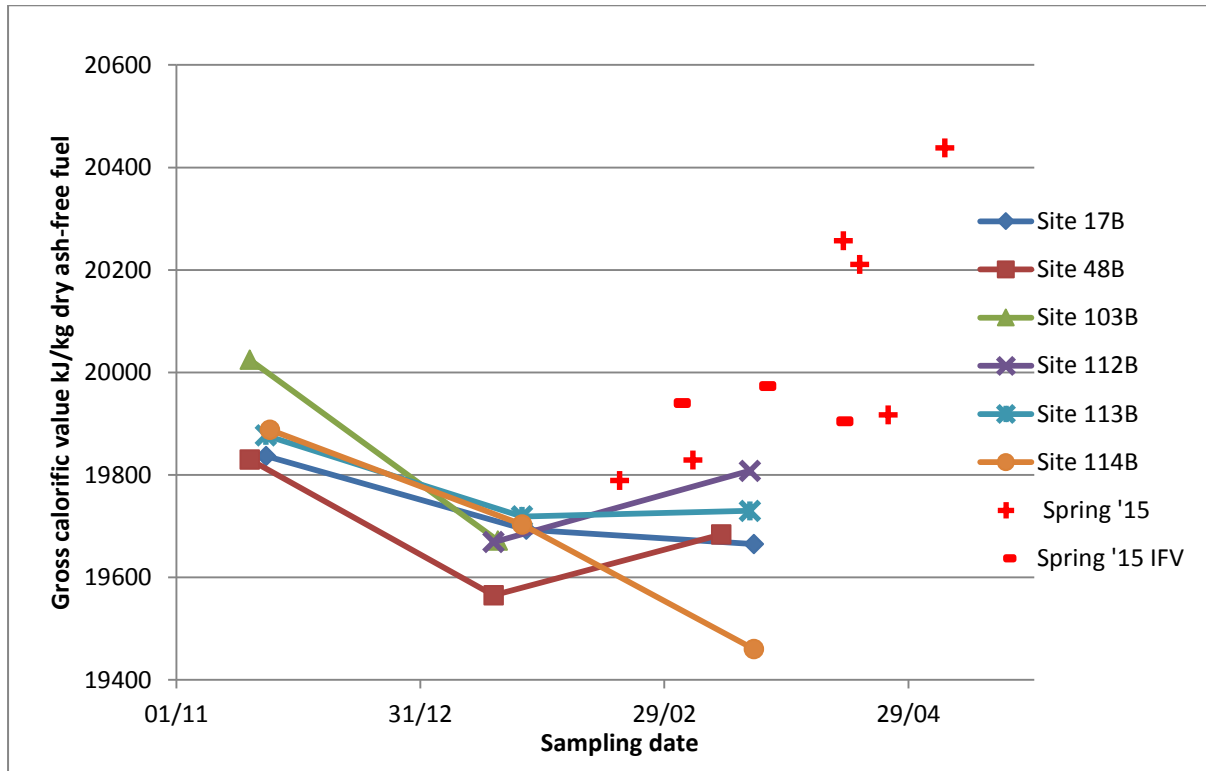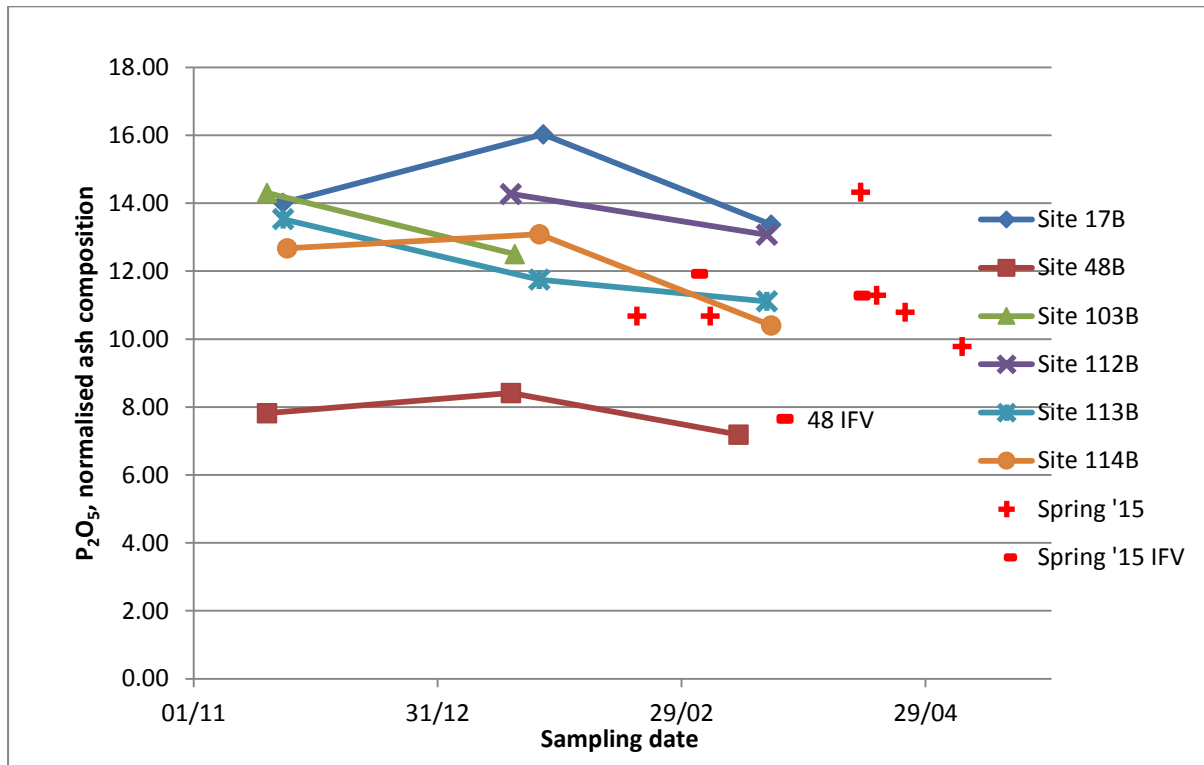
Figure 4-16: Changes in phosphate in willow SRC in spring 2016 plus values in red from 2015

4.2.3 Summary

- Only a few characteristics of willow SRC grown at six sites from north west to southern England showed statistically significant differences across three simulated harvesting times (mid-November, mid-January and mid-March) – GCV (DAF), chromium (d), $CaCO_3$ (na), K_2O (na), and P_2O_5 (na) - with the majority showing no difference. For the characteristics that did change, a variety of patterns was evident.

- Gross calorific values of willow SRC at all sites fell between mid-November and mid-January; by mid-March the GCV was either similar or had increased. Data from spring 2015 suggested that the increase in GCV may continue through to at least early May.

- There are several possible reasons for the lack of seasonal differences in willow SRC, especially in relation to *Miscanthus*, but the results are not unexpected. There is very limited literature to support seasonal changes in proximate and ultimate fuel properties or ash-forming elements (only GCV was significant in the present study) and although willows are well known as bio-accumulators of trace elements and heavy metals, the soils at sites studied were very low in concentrations of both.

- Our results suggest that willow SRC growers have considerable flexibility over harvesting times. This window should be limited to after leaf fall through to bud burst because inclusion of leaf material risks raising the moisture content, and ash, nitrogen, sulphur and chlorine levels considerably.

4.3 Study 7: The effect of variety on the feedstock characteristics of willow SRC in spring 2016

To recap, six varieties (Endurance, Tora, Terra Nova, Resolution, Sven, and Nimrod) of willow SRC were sampled at a range of sites from Northern Ireland, through Wales to eastern England (Aberystwyth, site 115; Rothamsted, site 116; Long Ashton, site 117; Loughall, site

118; and Brook Hall, site 119; see Figure 2-2). These sites spanned a wide range of environmental conditions to provide a robust test of the consistency of willow SRC feedstock characteristics across a range of varieties. Sampling at all sites was done within one week (end February/beginning of March 2016) to minimise the impact of sampling time on feedstock characteristics.

The REML analysis indicated that there was a significant difference between the varieties' response, averaged across sites, for approximately half of the feedstock characteristics, including the majority of proximate and ultimate fuel characteristics and normalised ash oxides. These results are presented for the feedstock characteristics of interest in Appendix 6 in the form of a standard set of simplified graphs in which a series of horizontal lines indicates that there was a significant difference between the varieties' response for that particular variable.

A further assessment of the *consistency* of the varieties' ranking across sites was done using Kendall's Coefficient of Concordance which is a measure of association between the rankings on the individual varieties (see Table 4-4). The most highly significant differences ($P<0.01$) in rankings were in moisture, net calorific value, carbon, nitrogen and $CaCO_3$ which are each plotted below using the same colour scheme to identify varieties to aid interpretation. In addition differences in the ranking of gross calorific value, copper, calcium, magnesium, manganese, K_2O, MgO and Na_2O were significant with *$P<0.05$ and >0.01*. These are plotted in Appendix 7 and summarised below.

In terms of moisture content, Endurance was consistently the lowest, with Tora, Resolution and Sven in the mid-range, with Terra Nova and Nimrod generally having the highest moisture contents (see Figure 4-17). As expected, the pattern of net calorific value was in essence the opposite of moisture so it is not graphed.

The carbon content was generally lowest in Sven followed by Resolution and highest in Nimrod with the other varieties intermediate (Figure 4-18).

Figure 4-17:   Moisture content and moisture content rankings of six willow SRC varieties at five sites across the UK harvested within one week in late February – early March 2016

Rankings of 1/A are lowest and 6/D are highest. Absolute values are shown in the chart below the table. An alphabetic code is used at Site 115 to emphasise that there were fewer varieties tested so a rank of 5 and 6 were not possible.

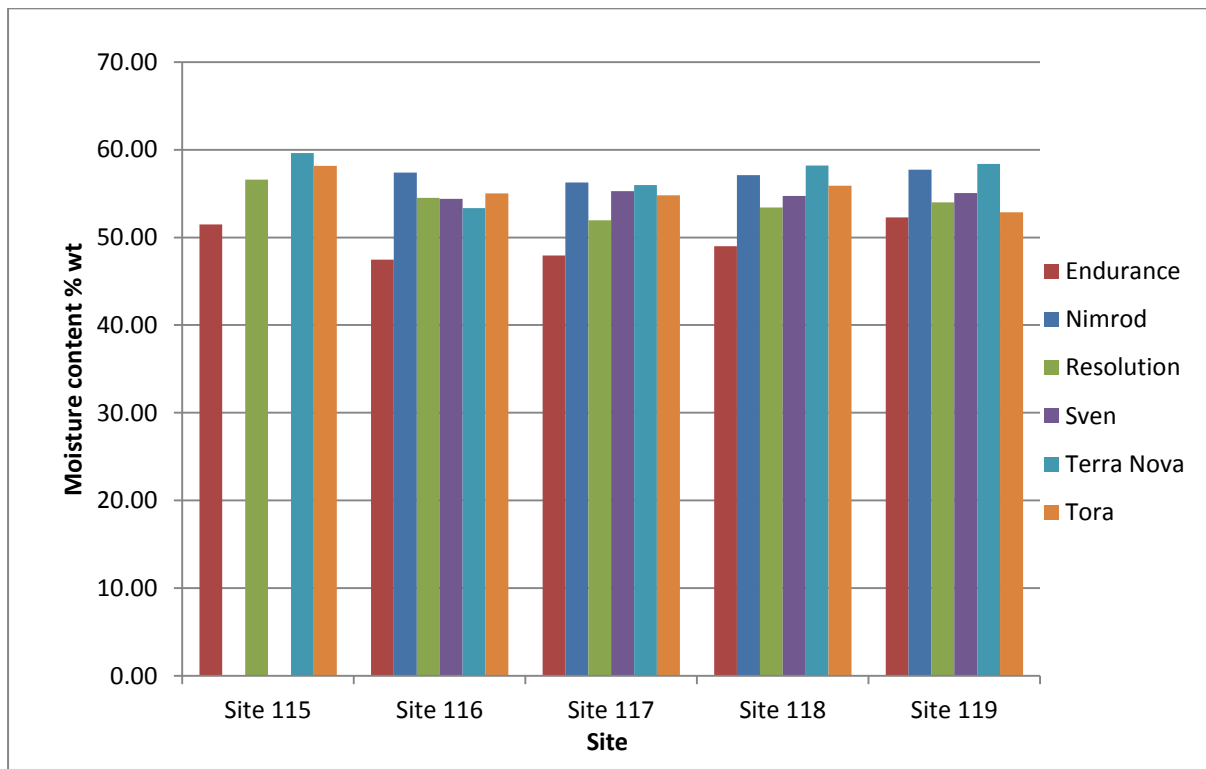| Site / Ranking	115	116	117	118	119
Endurance	A	1	1	1	1
Nimrod		6	6	5	5
Resolution	B	4	2	2	3
Sven		3	4	3	4
Terra nova	D	2	5	6	6
Tora	C	5	3	4	2

Table 4-4: **Analysis of varietal differences by feedstock characteristic, showing the concordance statistic and statistical probability of rankings across sites.**

Variable (basis of analysis)[†]	Statistic	Statistical significance[#]
Moisture (ar)	0.707	<0.01
Net calorific value (ar)	0.732	<0.01
Ash content (d)	0.606	NS
Volatile matter (DAF)	0.457	NS
Gross calorific value (DAF)	0.521	<0.05
Carbon (DAF)	0.750	<0.01
Hydrogen (DAF)	0.320	NS
Nitrogen (DAF)	0.668	<0.01
Sulphur (DAF)	0.490	NS
Chlorine (DAF)	0.538	NS
Barium (d)	0.403	NS
Beryllium (d)	0.427	N/A
Chromium (d)	NA	NA
Cobalt (d)	0.578	NS
Copper (d)	0.683	<0.05
Molybdenum (d)	0.517	N/A
Nickel (d)	NA	NA
Vanadium (d)	0.292	NS
Zinc (d)	0.644	NS
Antimony (d)	0.436	N/A
Arsenic (d)	0.250	N/A
Mercury (d)	0.333	N/A
Fluorine (d)	0.474	N/A
Bromine (d)	0.444	N/A
Selenium (d)	0.469	N/A
Cadmium (d)	0.327	NS
Lead (d)	0.417	NS
Aluminium (d)	0.064	NS
Calcium (d)	0.621	<0.05
Iron (d)	0.121	NS
Potassium (d)	0.193	NS
Magnesium (d)	0.563	<0.05
Manganese (d)	0.600	<0.05
Sodium (d)	0.229	NS
Phosphorous (d)	0.293	NS
Silicon (d)	0.088	NS
Titanium (d)	0.114	NS
Al_2O_3 (na)	NA	NA
BaO (na)	0.802	NS
$CaCO_3$ (na)	0.636	<0.01
Fe_2O_3 (na)	NA	NA
K_2O (na)	0.631	<0.05
MgO (na)	0.574	<0.05
Mn_3O_4 (na)	0.605	NS
Na_2O (na)	0.521	<0.05
P_2O_5 (na)	0.500	NS
SiO_2 (na)	NA	NA
TiO_2 (na)	0.266	N/A
Alkali index	0.221	NS

[†]basis of analysis; ar = as received fuel; d = dry fuel; DAF = dry; ash-free fuel; na= normalised ash; [#] NS=not significant; NA=not analysed; N/A = values below or close to limit of detection so trends are not interpreted, <0.05=<5% and <0.01=<1%)

Figure 4-18: **Carbon content and carbon content rankings of six willow SRC varieties at five sites across the UK harvested within one week in late February – early March 2016**

Rankings of 1/A are lowest and 6/D are highest. Absolute values are shown in the chart below the table. An alphabetic code is used at Site 115 to emphasise that there were fewer varieties tested so a rank of 5 and 6 were not possible.

| Site / Ranking	115	116	117	118	119
Endurance	C	3	5	5	3
Nimrod		6	6	6	6
Resolution	A	4	1.5	2	1
Sven		1	1.5	1	2
Terra nova	D	2	4	4	5
Tora	B	5	3	3	4
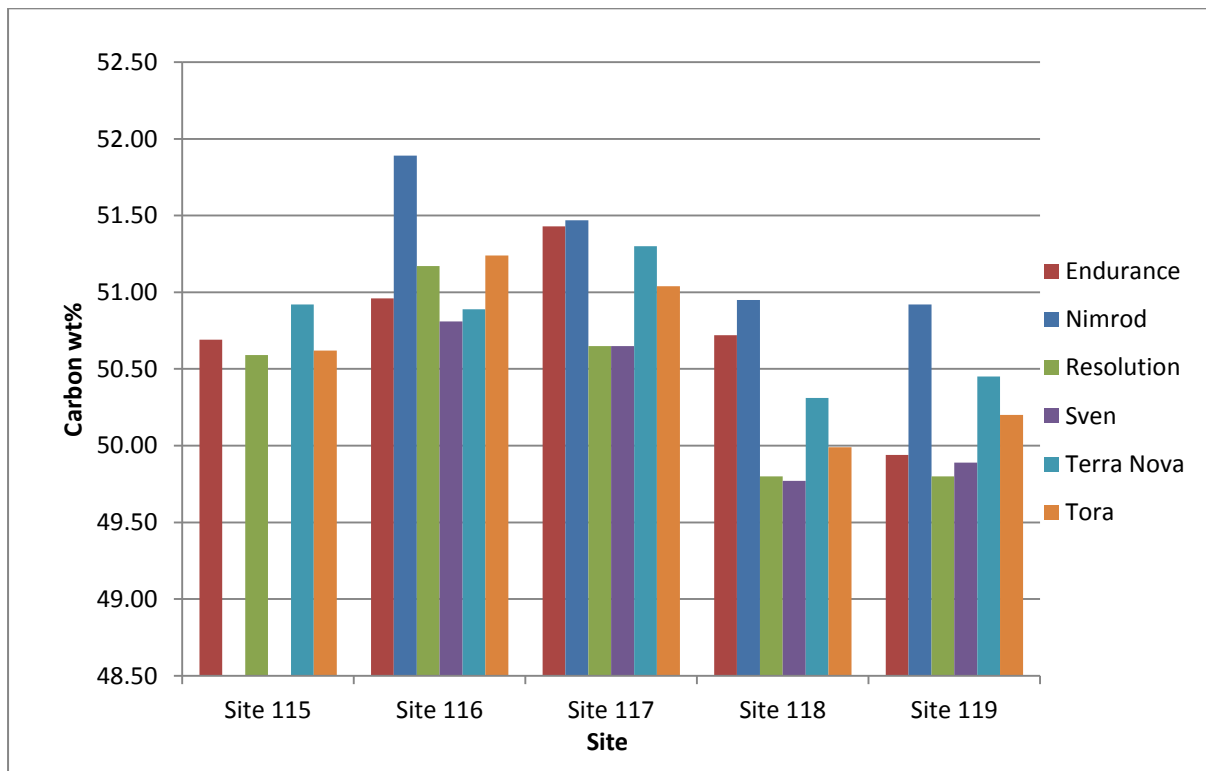
Turning to nitrogen content (Figure 4-19), Resolution generally had low levels whereas Nimrod, Terra Nova and Endurance generally had high nitrogen levels. Sven was intermediate across the four sites where it was present. Tora had the lowest levels at several sites but was in the mid-range at two sites.

Figure 4-19: **Nitrogen content and nitrogen content rankings of six willow SRC varieties at five sites across the UK harvested within one week in late February – early March 2016**

Rankings of 1/A are lowest and 6/D are highest. Absolute values are shown in the chart below the table. An alphabetic code is used at Site 115 to emphasise that there were fewer varieties tested so a rank of 5 and 6 were not possible.

| Site / Ranking	115	116	117	118	119
Endurance	D	5.5	6	4	4
Nimrod		5.5	4	5.5	5
Resolution	C	1	2	2	1
Sven		3	3	3	2
Terra nova	B	2	5	5.5	6
Tora	A	4	1	1	3
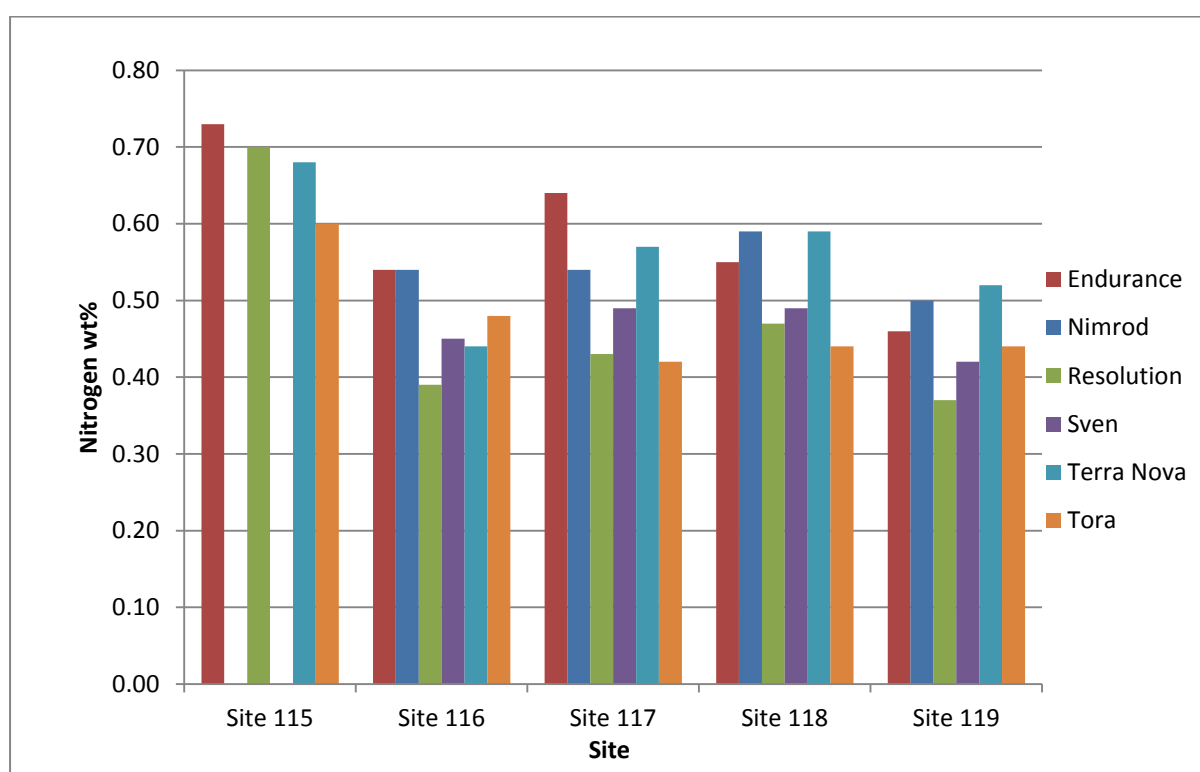
Resolution had low levels of $CaCO_3$ (Figure 4-20) followed by Sven. The other varieties (Endurance, Tora, Terra Nova and Nimrod) had rather variable rankings depending on the site and each had the highest level at one site.

Figure 4-20: Calcium carbonate content in ash and calcium carbonate content in ash rankings of six willow SRC varieties at five sites across the UK harvested within one week in late February – early March 2016

Rankings of 1/A are lowest and 6/D are highest. Absolute values are shown in the chart below the table. An alphabetic code is used at Site 115 to emphasise that there were fewer varieties tested so a rank of 5 and 6 were not possible.

Site / Ranking	115	116	117	118	119
Endurance	C	6	5	4	3
Nimrod		5	2	5	6
Resolution	A	1	1	2	1
Sven		2	3	1	2
Terra nova	D	4	4	6	5
Tora	B	3	6	3	4
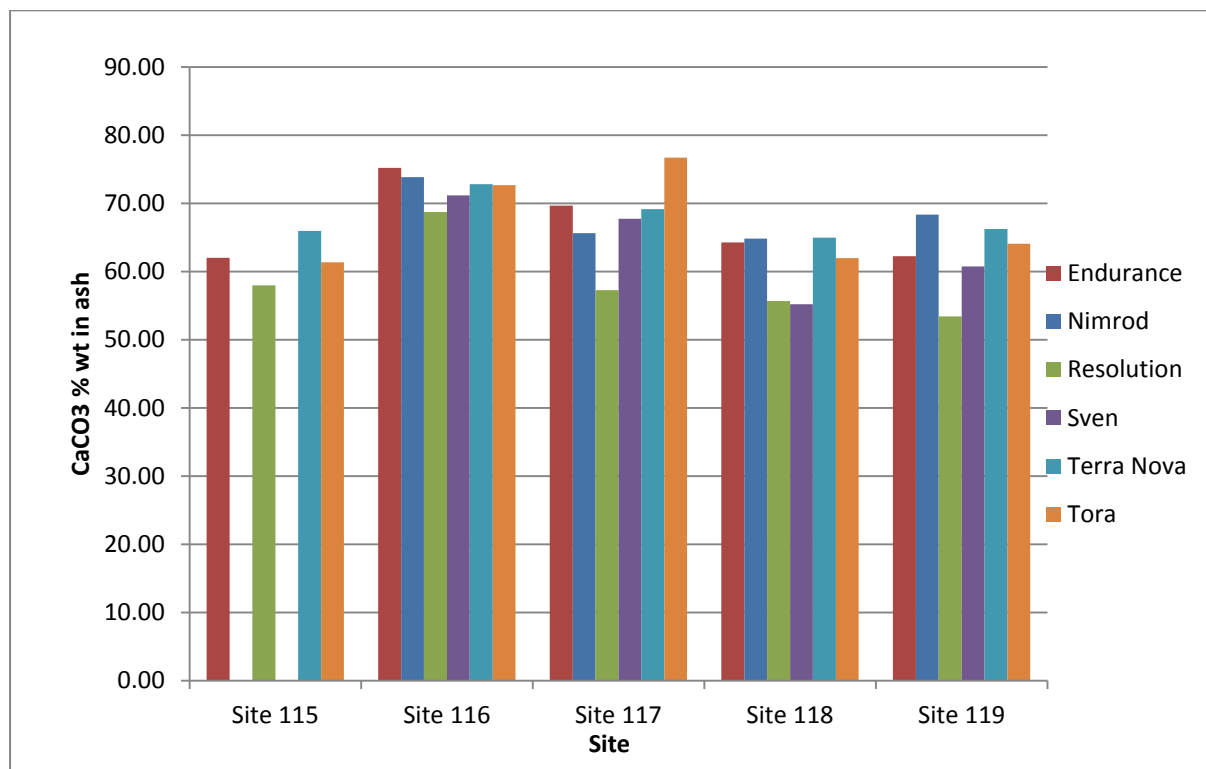
Summarising the main points to emerge from the rankings that were significant with *P<0.05* and *>0.01* (see Appendix 7):

- Nimrod had consistently high gross calorific value
- Tora had consistently low levels of copper at the three sites where rankings could be statistically evaluated (sites 116, 117, and 118); Nimrod generally had high levels of copper
- Resolution, followed by Sven, had consistently low levels of calcium; the other four varieties were more variable and each had the highest levels at one site

- Endurance had high levels of magnesium but generally the lowest levels of manganese
- Resolution had high levels of K_2O in the ash whereas Terra Nova generally had the lowest levels
- Resolution had high levels of MgO in the ash as did Endurance
- Resolution also high levels of Na_2O in the ash.

It is worth drawing attention to the results for ash. During the quality assurance of data, one very high ash value (3% for Endurance at Site 117) was excluded and consequently the statistical analysis of the varieties' ash content ranking could not be completed because there were insufficient data points. A visual assessment of the data however suggest that Resolution had a consistently low ash content whereas Nimrod generally had a high ash content and Terra Nova often had a high ranking (see final graph in Appendix 7).

Considering the feedstock parameters where the varieties tended to have a consistent ranking across the sites, it is apparent that Resolution had the lowest carbon, nitrogen and calcium. Although there was not a full data set for ash content, there was a suggestion that Resolution also had a low ash content. These properties would generally be advantageous but there is also a tendency for Resolution to have the highest concentrations of K_2O, MgO and Na_2O in the ash which would have detrimental impacts. K_2O and Na_2O are key concerns for plant corrosion and slagging; they may also result in the formation of fine particulate matter which is an issue for emissions and amine-based carbon capture processes. Furthermore K_2O is a poison for NO_x reduction catalysts. Thus the choice of Resolution would have positive and negative implications and it must also be remembered that the yield of the different varieties would be important.

4.3.1 Summary

- There was a certain degree of consistency in willow variety properties across sites from Northern Ireland to Southern England, with approximately 40% of the parameters analysed showing statistically consistent rankings for the varieties tested (Endurance, Nimrod, Resolution, Sven, Terra Nova, and Tora). Considering the results as a whole no variety combined the best ranking in all parameters.
- For the majority of parameters however, there was not a consistent ranking. Volatile matter, sulphur and chlorine content for example did not show consistent rankings and neither did the alkali index.

4.4 Study 8: The impact of storage after baling on *Miscanthus* composition in summer 2016

### 4.4.1 Selection of storage treatments

The results from Study 8 are summarised in Appendix 10 and the key points are given here since they determined the choice of storage types and durations. The majority (15) of the 20 growers surveyed stored their bales in either a fully enclosed barn, or a partially enclosed shed (3 sides and a roof), with three growers storing bales outside under sheets, and only one grower interviewed storing bales outside with no sheeting. One other grower did not require any storage as bales left the farm straight away (see Figure 4-21).

Figure 4-21: Type of on-farm storage used by *Miscanthus* growers in the UK
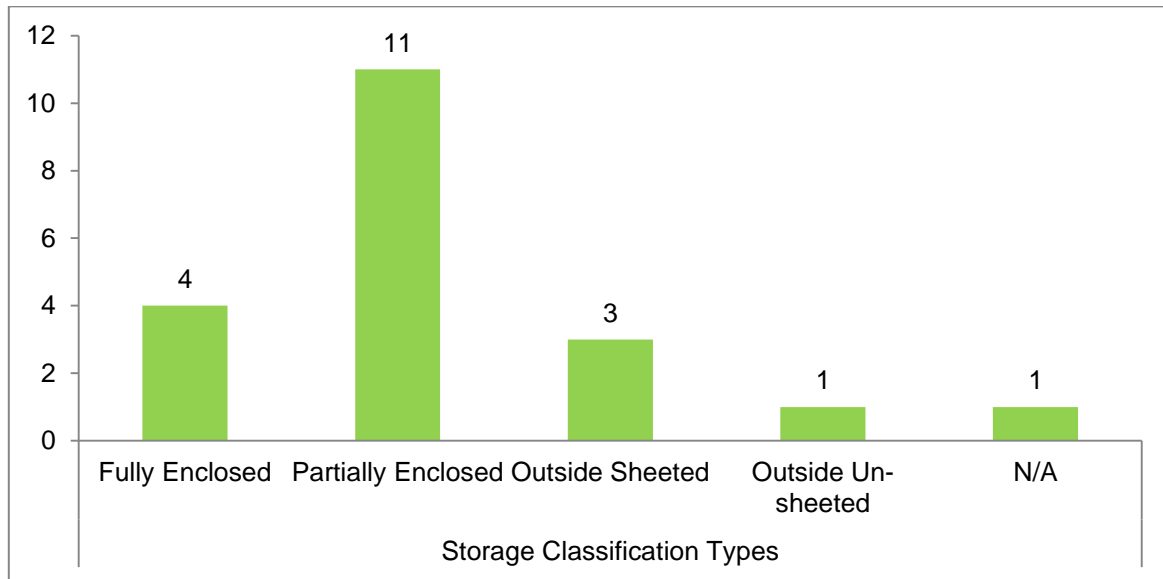
Storage time varied between growers (see Figure 4-22) and also year on year, but the total storage periods were broadly similar across all the growers.

Figure 4-22: Duration of on-farm storage used by *Miscanthus* growers
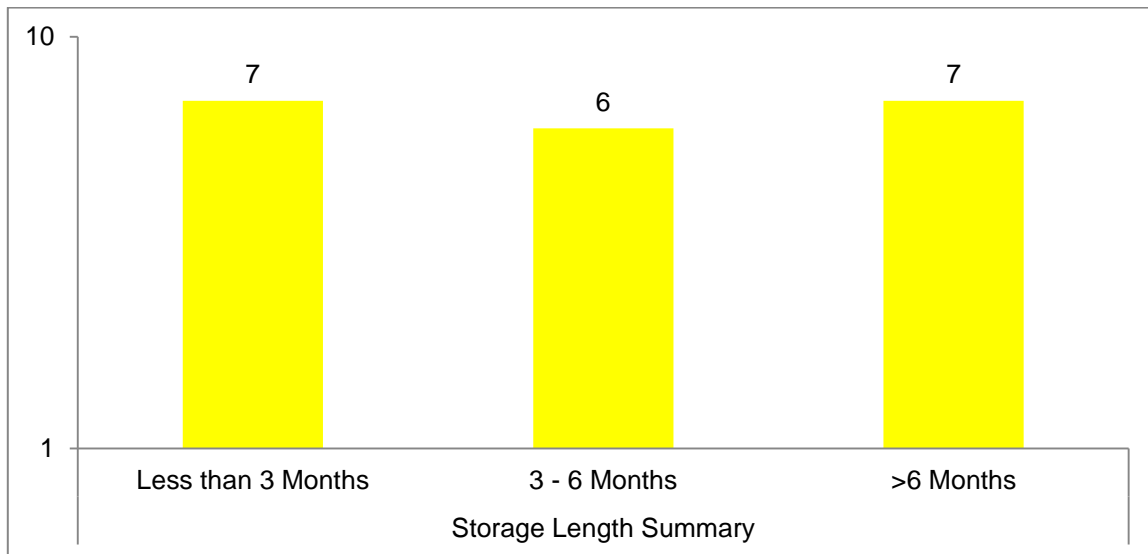
As a result of the findings from the questionnaire, the four storage types used in Study 8 were: storage outside with no cover; storage outside with waterproof sheeting protecting the top of the stack of bales and the sides of the upper two/three bale layers; storage inside in a building with open sides providing roof cover only; and storage inside in a fully enclosed building. The maximum storage duration was set at five months, giving six sampling times in total; longer storage was not possible because of the length of the ETI contract but it was thought that any trends would be evident within five months.

4.4.2 Impact of *Miscanthus* storage type and duration

As explained in section 2.2.4, one part of the stack used for each storage type was sampled only at the start and end of the experiment to replicate normal commercial practice where bales would not be disturbed; the other part of each stack was opened up at monthly intervals

to ensure representative sampling throughout the stack. The statistical analysis showed that there were no significant differences between the repeated sampling and the start/end sampling across any of the variables (see Table 4-5). This indicates that the results on the changes through time are applicable to normal operational conditions.

Table 4-5:   Changes in mean absolute values over the total storage period relative to starting value.

The two sampling procedures are shown, i.e. sampled at the start and end of the storage period only cf sampled at monthly intervals and the statistical significance of the difference in sampling procedure.

Variable (basis of analysis)†	Mean change relative to starting value		Statistical significance*
	Start/End Sampling	Repeated Sampling	
Moisture (ar)	3.29	4.03	0.249
Net calorific value (ar)	-793	-907	0.395
Ash content (d)	0.63	0.43	0.239
Volatile matter (DAF)	-1.26	-1.01	0.557
Gross calorific value (DAF)	-38.25	-46.50	0.883
Carbon (DAF)	0.56	0.64	0.746
Hydrogen (DAF)	-0.05	-0.08	0.510
Nitrogen (DAF)	-0.06	-0.06	0.911
Sulphur (DAF)	0.03	0.02	0.730
Chlorine (DAF)	0.09	0.06	0.107
Barium (d)	-0.51	0.65	0.254
Beryllium (d)	0.56	0.62	0.638
Cobalt (d)	0.05	-0.38	0.333
Molybdenum (d)	0.07	0.02	0.508
Nickel (d)	NA	NA	NA
Vanadium (d)	0.51	0.56	0.512
Zinc (d)	1.84	0.11	0.723
Bromine (d)	9.22	5.63	0.090
Cadmium (d)	-0.017	0.000	0.189
Lead (d)	0.10	-0.23	0.338
Aluminium (d)	10.55	15.40	0.417
Calcium (d)	258	258	0.999
Iron (d)	3.27	13.22	0.260
Potassium (d)	-1410.25	-1937.75	0.245
Magnesium (d)	69.88	5.23	0.067
Manganese (d)	47.78	16.24	0.084
Sodium (d)	52.74	34.13	0.409
Phosphorous (d)	263.9	149.6	0.327
Silicon (d)	1723	950	0.338
Titanium (d)	-1.52	-2.17	0.768
Al_2O_3 (na)	0.06	0.16	0.187
BaO (na)	-0.01	0.01	0.135
$CaCO_3$ (na)	2.22	2.85	0.735
Fe_2O_3 (na)	-0.03	0.09	0.230
K_2O (na)	-14.22	-14.34	0.903
MgO (na)	-0.16	-0.21	0.937
Mn_3O_4 (na)	0.30	0.07	0.228
Na_2O (na)	0.18	0.15	0.893
P_2O_5 (na)	1.59	1.35	0.923
SiO_2 (na)	13.05	9.97	0.503
TiO_2 (na)	-0.02	-0.05	0.543
Alkali index	-0.09	-0.12	0.282

† basis of analysis; ar = as received fuel, d = dry fuel, DAF = dry, ash-free fuel, na= normalised ash; NA = not applicable; * no difference is significant with a probability of less than 1 in 20 *(P<0.05)*

The type of storage had a statistically significant effect on ash, nitrogen and sulphur contents of the samples and the impact on gross calorific value was close to being significant (*P=0.056); see **Error! Not a valid bookmark self-reference.***. Analysis of monthly samples showed that storage duration had a significant impact on nitrogen and that there was an interaction between storage type and duration in the case of ash. Bromine was significantly influenced by storage type, duration and their interaction. Zinc and calcium contents were also significantly affected by the type of storage although it was not possible to say if storage duration had an influence or not. Where there was a significant treatment effect values generally increased (ash content, sulphur, zinc, bromine, calcium) while the levels of only nitrogen decreased over the five months. There were no significant differences in storage types or timings across any of the major oxides.

The overall changes during storage *where there has been no effect of storage type or duration* are shown on the right of (**Error! Not a valid bookmark self-reference.**). These are described briefly in the following paragraph, after which the significant treatment effects of storage type and duration are examined in more detail.

Storage for 5 months, even for variables that were not significantly affected by either the type or duration of storage, had a significant impact on approximately 40% of the variables (**Error! Not a valid bookmark self-reference.**). Significant changes were found in all of the main groupings: proximate and ultimate analysis, trace elements, ash forming elements including the alkali index, and ash oxides. In the majority of cases values increased - significant increases were observed in: moisture (see Figure 4-23), chlorine (Figure 4-24), beryllium, vanadium, aluminium, manganese, sodium, phosphorus, silicon, plus the oxides of aluminium, calcium, and silicon. Significant decreases were observed in net calorific value, volatile matter, hydrogen, potassium, potassium oxide and the alkali index.

Table 4-6:   Analysis of the impact of storage type and where possible the effect of storage duration and the interaction between storage type and duration.

Grey cell fill denotes where there were no scheduled monthly analyses. Where the effect of storage treatment was not significant, the three columns on the right show the statistical significance of the difference between the initial and final values and the change in absolute terms.

Variable (basis of analysis)[†]	Significance of storage type*	Significance of storage duration	Significance of type x duration interaction	Statistical significance of difference between start and end	Estimated change relative to initial condition	Estimated standard error of change
Moisture (ar)	NS	NS	NS	0.000	3.69	0.30
Net calorific value (ar)	NS	NS	NS	0.000	-791	56.5
Ash content (d)	<0.01	NS	<0.05	Treatment effect	NA	NA
Volatile matter (DAF)	NS	NS	NS	0.002	-1.03	0.20
Gross calorific value (DAF)	0.056	NS	NS	NS	-21.5	28.9
Carbon (DAF)	NS	NS	NS	NS	0.15	0.18
Hydrogen (DAF)	NS	NS	NS	0.025	-0.07	0.02
Nitrogen (DAF)	<0.01	<0.01	NS	Treatment effect	NA	NA
Sulphur (DAF)	<0.05	NS	NS	Treatment effect	NA	NA
Chlorine (DAF)	NS	NS	NS	0.000	0.07	0.01
Barium (d)	NS			NS	-0.01	0.47
Beryllium (d)	NS			0.000	0.58	0.05
Cobalt (d)	NS			NS	-0.16	0.20
Molybdenum (d)	NS			NS	0.06	0.03
Nickel (d)	NA			NA	NA	NA
Vanadium (d)	NS			0.002	0.54	0.02
Zinc (d)	<0.05			Treatment effect	NA	NA
Bromine (d)	<0.01	<0.01	<0.01	Treatment effect	NA	NA
Cadmium (d)	NS			NS	-0.01	0.01
Lead (d)	NS			NS	-0.04	0.16
Aluminium (d)	NS			0.002	12.97	2.74
Calcium (d)	<0.05			Treatment effect	NA	NA
Iron (d)	NS			NS	7.53	4.07
Potassium (d)	NS			0.000	-1674	214
Magnesium (d)	NS			NS	32.94	18.11
Manganese (d)	NS			0.010	32.01	9.23
Sodium (d)	NS			0.004	43.4	10.3
Phosphorous (d)	NS			0.009	199	52.9
Silicon (d)	NS			0.009	1336	373
Titanium (d)	NS			NS	-1.80	0.95
Al_2O_3 (na)	NS			0.020	0.11	0.04
BaO (na)	NS			NS	0.00	0.00
$CaCO_3$ (na)	NS			0.019	2.58	0.81
Fe_2O_3 (na)	NS			NS	0.02	0.05
K_2O (na)	NS			0.000	-14.28	0.45
MgO (na)	NS			NS	-0.19	0.23
Mn_3O_4 (na)	NS			NS	0.18	0.09
Na_2O (na)	NS			NS	0.16	0.10
P_2O_5 (na)	NS			NS	1.45	1.08
SiO_2 (na)	NS			0.001	11.51	2.09
TiO_2 (na)	NS			NS	-0.03	0.02
Alkali index	NS			0.000	-0.10	0.01

† basis of analysis; ar = as received fuel, d = dry fuel, DAF = dry, ash-free fuel, na= normalised ash.
NA = not applicable. * the smaller the value the less likely the difference is to have occurred by chance alone so it is more likely to be a real effect; NS means that the difference was not significant with a probability of less than 1 in 20.

Figure 4-23: Changes in moisture content during storage. There was a significant increase but not a significant effect of storage type or duration.
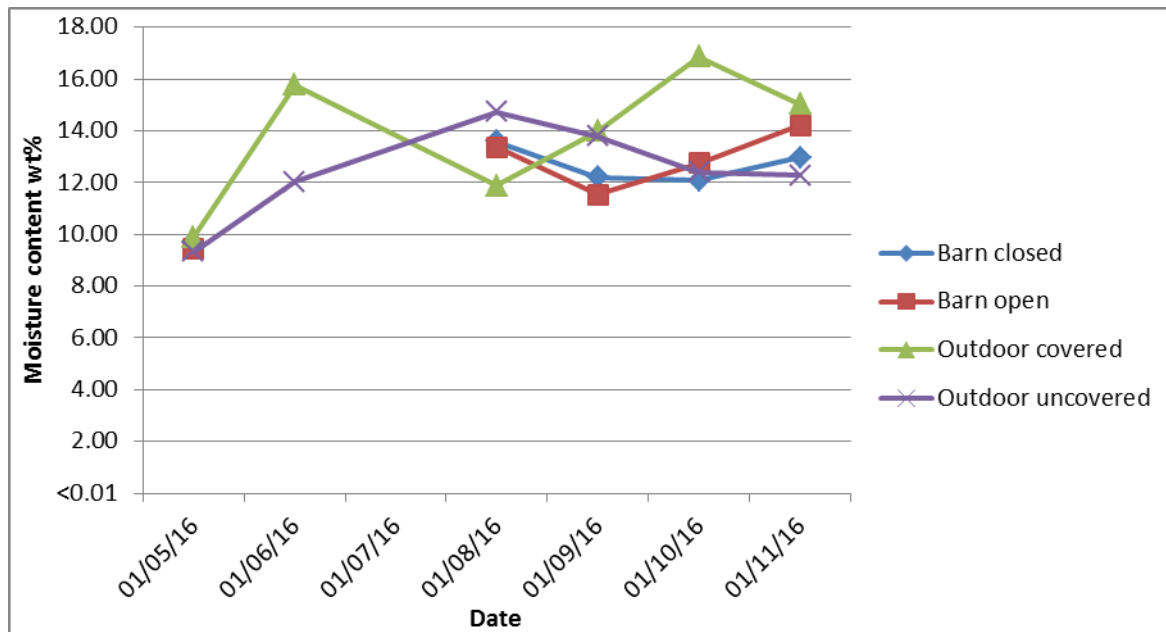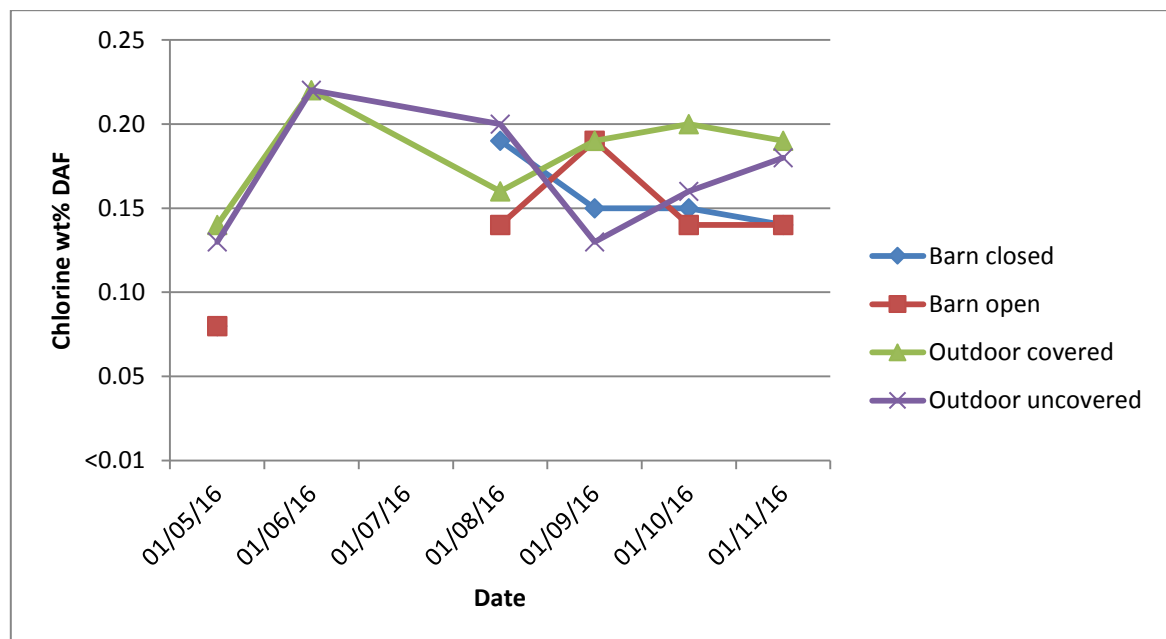

Figure 4-24: Changes in chlorine concentration during storage. There was a significant increase but not a significant effect of storage type or duration.

There were significant effects of storage regime ($P < 0.001$) and the interaction between storage regime and time ($P = 0.017$) for the ash percentage data. In all cases, ash percentages of samples collected following storage tended to be higher than the values of samples collected in May at the beginning of the storage period line (see Figure 4-25), i.e. all points are above the 0 line which shows no change. In addition the barn-stored bales had higher ash values towards the start of the experiment that then declined whereas outdoor samples started lower and increased towards the end of the experiment. For the barn-stored bales the November levels of ash were about 50% higher than the May values and the bales stored

outdoors were approximately 20% higher in November compared to May. To aid interpretation, the best fit model was applied to the original May data in
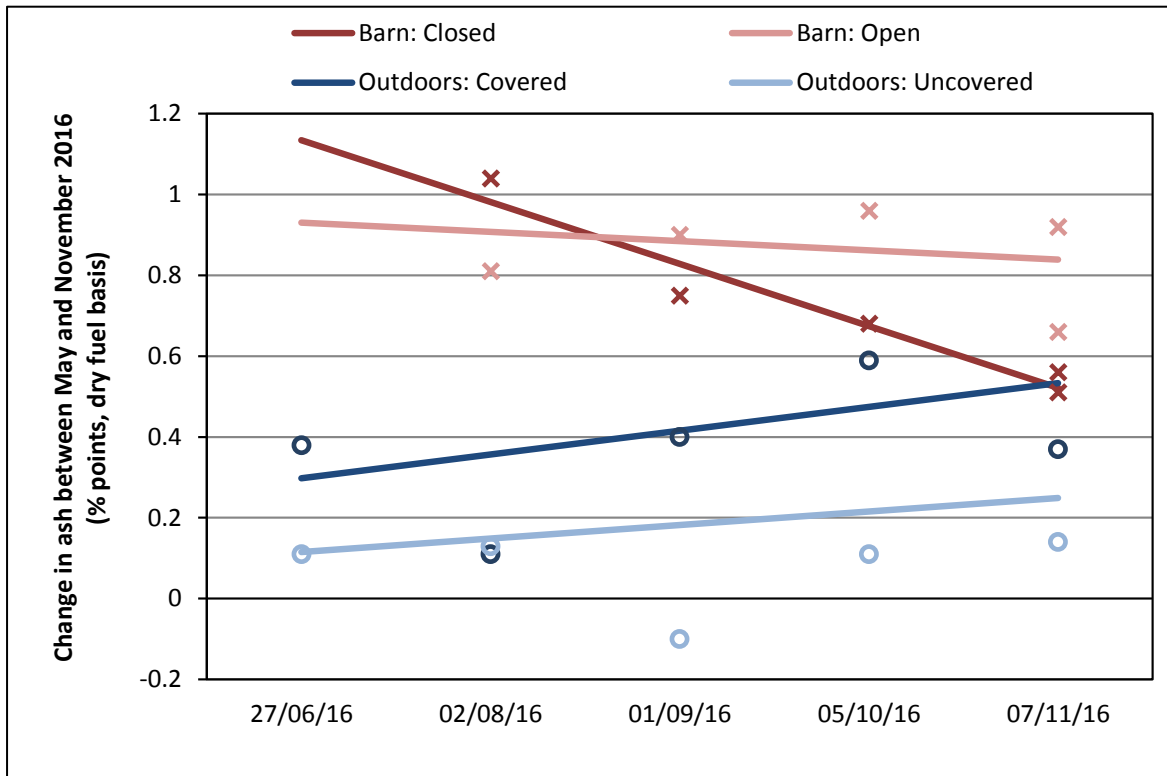
Figure 4-26. This showed ash content peaking in the barn data in the earlier months, but declining by the end of the experiment to be similar to the outdoor data.

Figure 4-25: **Change in ash content between May and November 2016 relative to the first assessment on 17th May (percentage point difference)**
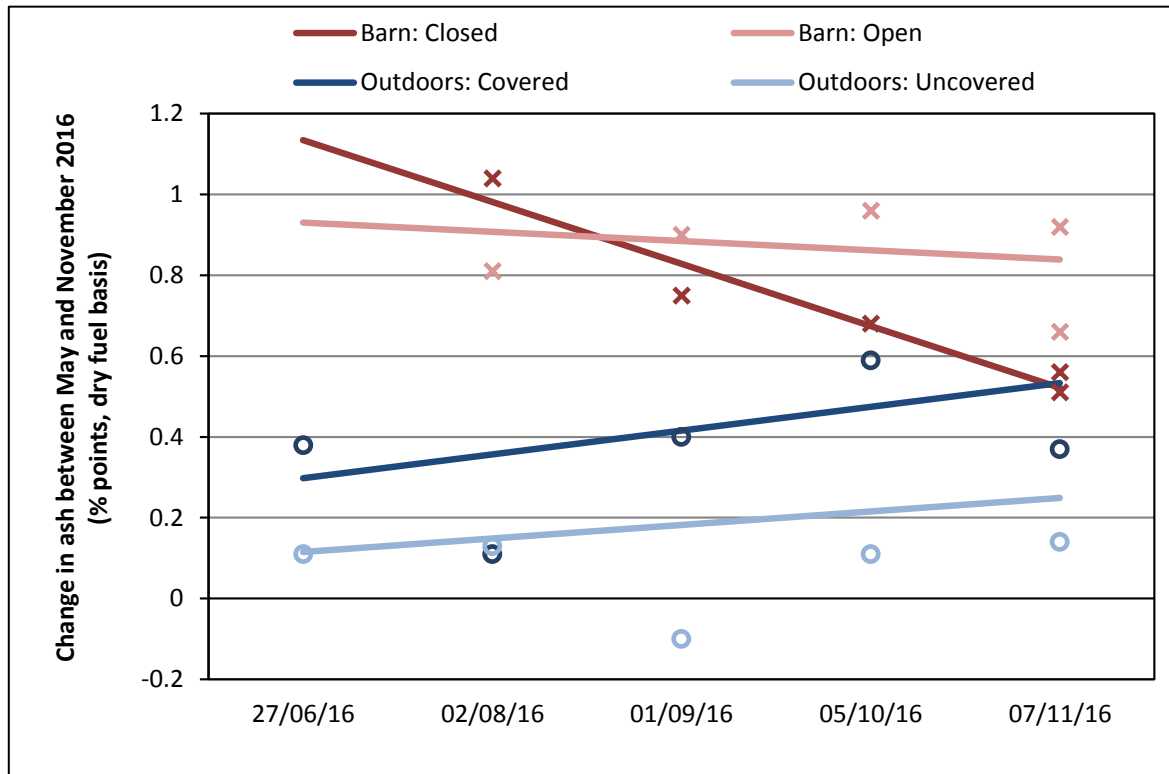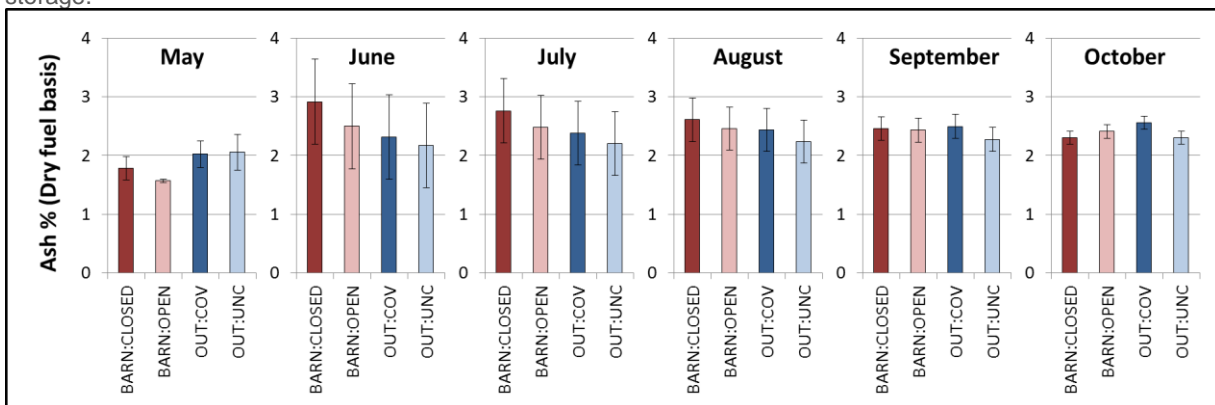

Figure 4-26: **Predicted change in ash content applied to original May data and projected through to October (monthly).**

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

There were significant additive effects of storage type ($P = 0.005$) and time ($P < 0.001$) for the percentage of nitrogen in the samples. In all four storage types, nitrogen declined through time, with a similar slope of -0.019% per month; on average nitrogen contents were 15% lower in November compared to May. Although the slopes were not significantly different, outdoors uncovered samples showed a greater absolute decline in nitrogen from the May values than the other three storage types (see Figure 4-27). To aid interpretation, the best fit model is applied to the original May data in

Figure 4-28. This shows the larger absolute change in the outdoors uncovered data; this treatment had the highest nitrogen content at the start of the experiment but the lowest by the end of the experiment.

Figure 4-27: Change in nitrogen content (as ash-free dry fuel) between May and November 2016 relative to the first assessment on 17th May (percentage point difference).
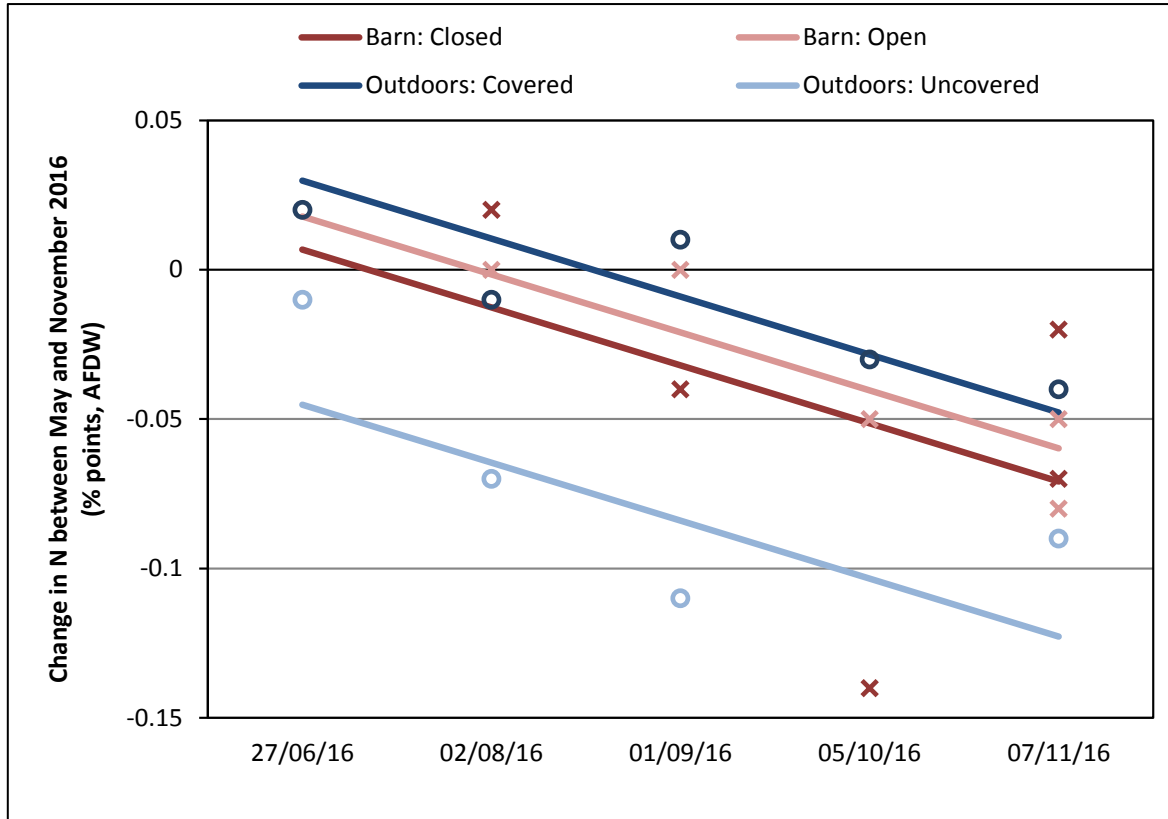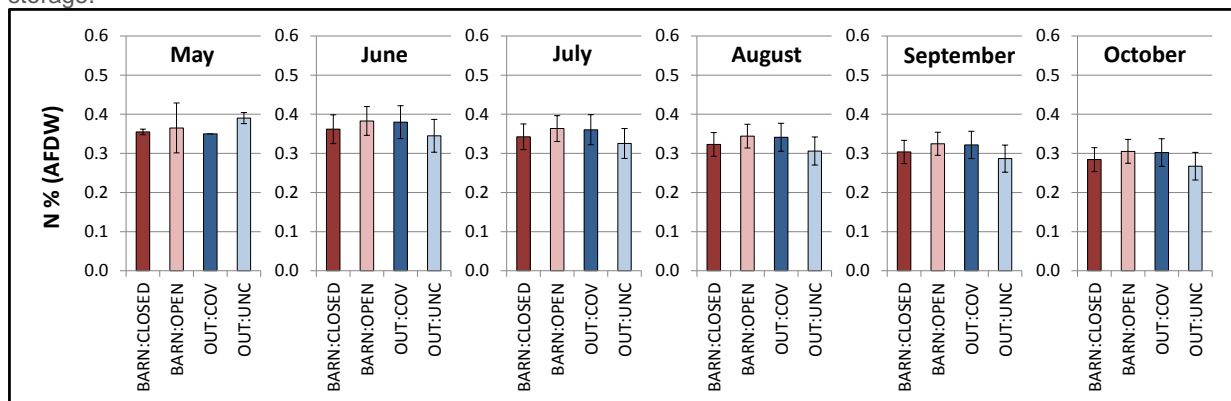

Figure 4-28: Predicted change in nitrogen content (as ash-free dry fuel) applied to original May data and projected through to October (monthly).

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

There were significant effects of storage type ($P = 0.043$) on the percentage of sulphur in the samples with an increase of approximately ~40%-150% in all samples between May and November. Those samples stored in barns tended to have larger increases in sulphur contents

than the outdoors samples (see Figure 4-29). Although there was an overall significant difference, it was not possible to determine pairwise significant differences, mostly due to the small sample size (as seen by the large error bars in Figure 4-29). To aid interpretation, the best fit model was applied to the original May data in Figure 4-30. This shows that sulphur contents tended to be lower in the barn data at the start of the experiment in May, but by the end of the experiment, the larger increases in the barn data resulted in similar sulphur contents across the four treatments.

Figure 4-29: **Mean change in sulphur content (as dry ash-free fuel) between May and November 2016 relative to the first assessment on 17th May (percentage point difference).**

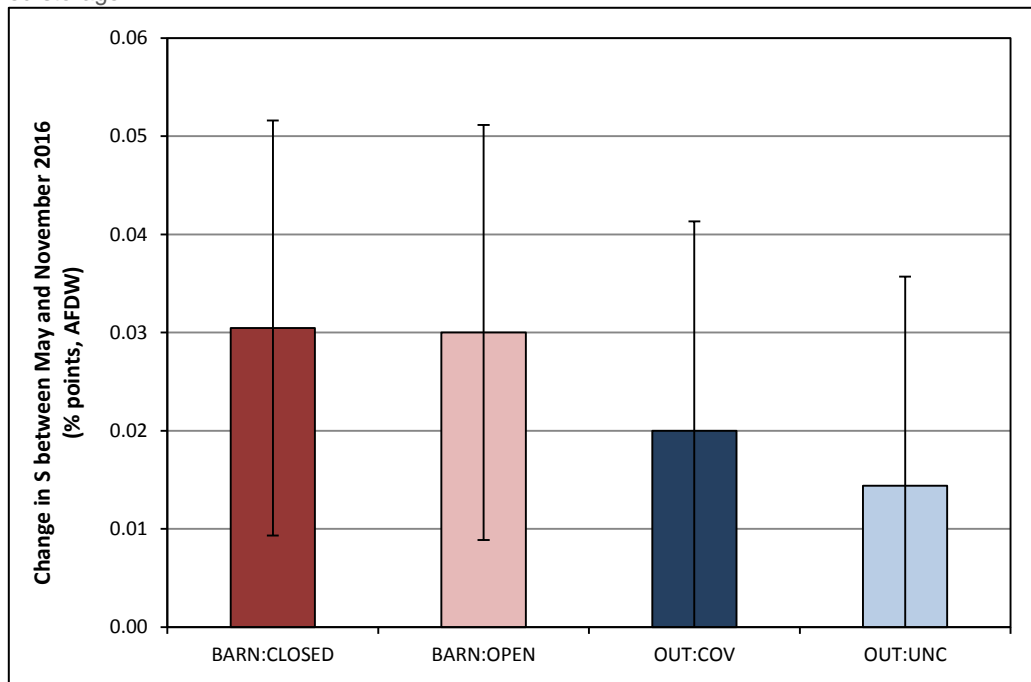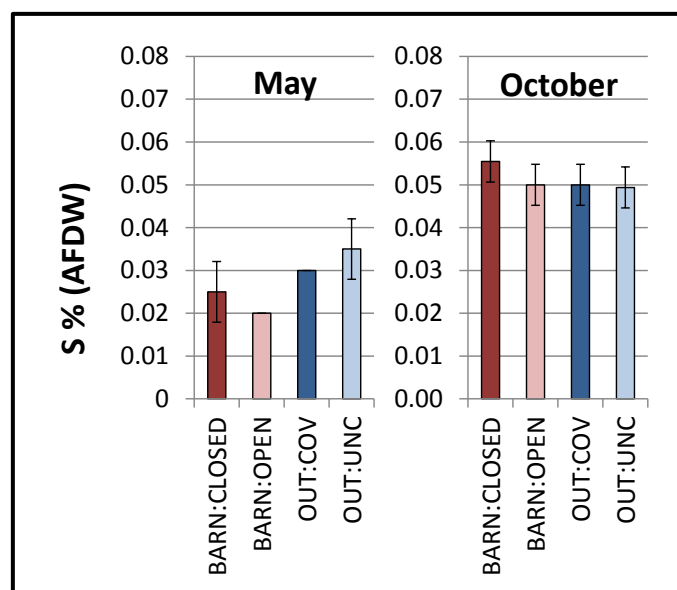Error bars = 95% confidence intervals. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

Figure 4-30: **Predicted change in sulphur content (as ash-free dry fuel) applied to original May data and projected through to October.**

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

There were significant effects of storage type ($P = 0.046$) on zinc content in the samples. Those samples stored in barns tended to show increases in zinc content (by approximately 50% over the duration of the storage) whereas the outdoors samples tended to show decreases (of approximately 20% see Figure 4-31). Although there was an overall significant difference, it was not possible to determine pairwise significant differences, mostly due to the small sample size (as seen by the large error bars in Figure 4-31). To aid interpretation, the best fit model is applied to the original May data in

Figure 4-32. This shows that at the start of the experiment, zinc contents were lower in the barn data, but by the end of the experiment, zinc contents were higher in these treatments.

Figure 4-31: Mean change in zinc content between May and November 2016 relative to the first assessment on 17th May.

Error bars = 95% confidence intervals. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.
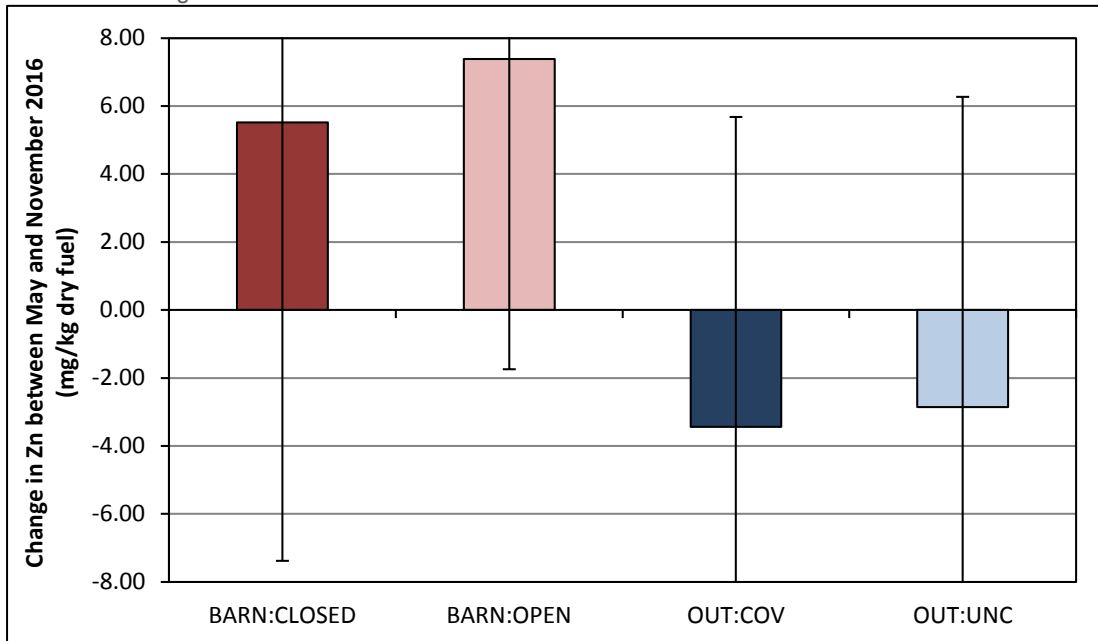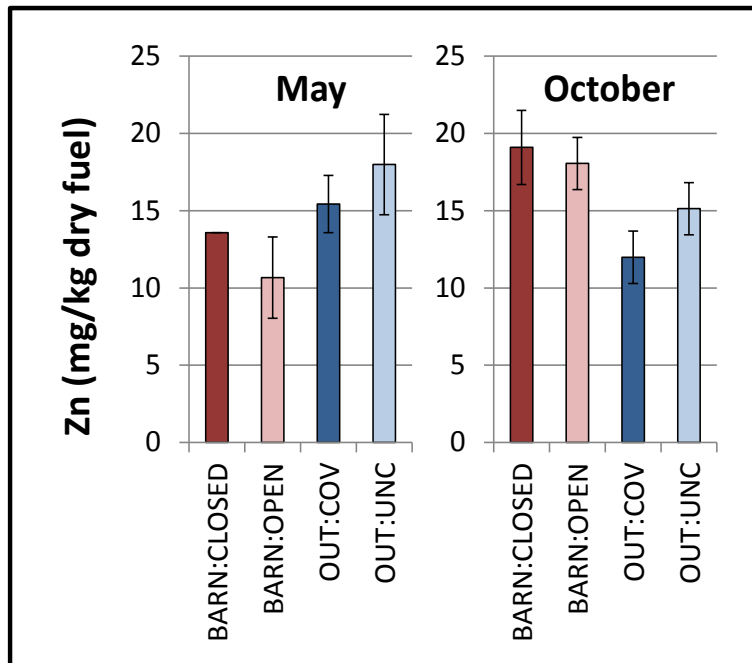

Figure 4-32: Predicted change in zinc content applied to original May data and projected through to October.

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

There were significant effects of storage type ($P < 0.001$), duration ($P < 0.001$) and the interaction between storage type and duration ($P = 0.002$) on bromine, shown in Figure 4-33; by the end of the experimental storage all types of storage had resulted in an increase in bromine. Outdoors uncovered samples showed a large drop in bromine content in June versus

May, with a steep incline through time, such that bromine content had increased (versus May) by the end of the experiment. Other storage types showed less steep inclines but from a positive base; by the end of the experiment, changes were similar across three out of the four storage types. To aid interpretation, the best fit model is applied to the original May data in Figure 4-34. The drop in bromine content in June, followed by a rapid increase, is clearly shown in the outdoors uncovered data.

Figure 4-33: **Change in bromine content between May and November 2016 relative to the first assessment on 17th May2016.**
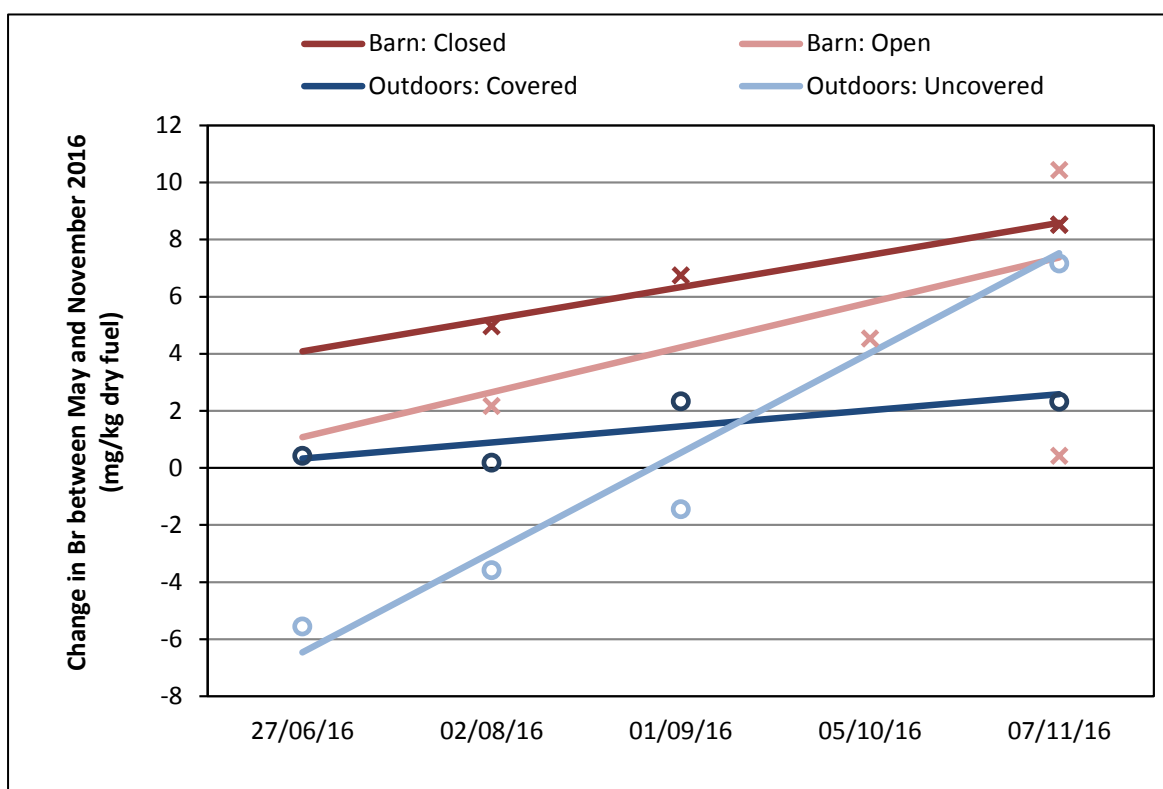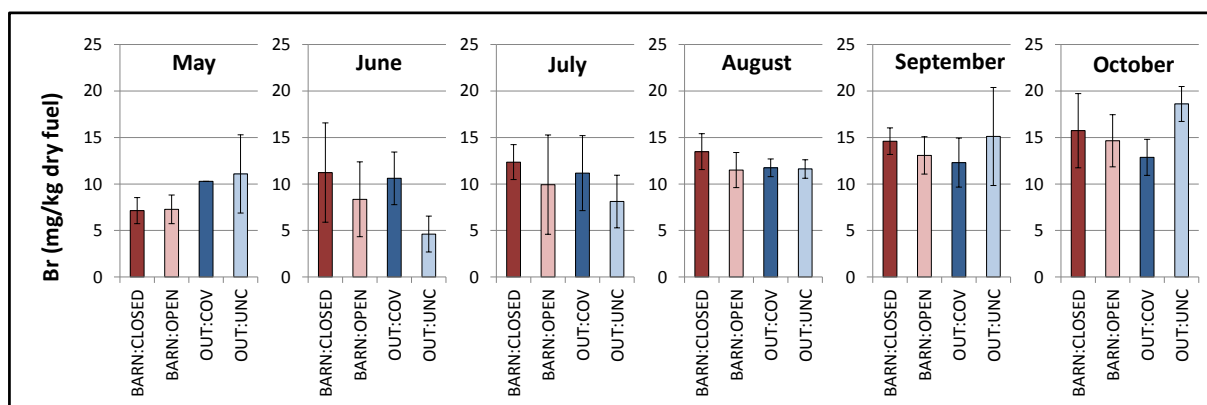

Figure 4-34: **Predicted change in bromine content applied to original May data and projected through to October (monthly). Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.**

Calcium contents rose by about one third during storage but there were significant effects of storage type (p = 0.016) on calcium content in the samples. Post-hoc tests supported this

result, indicating that calcium levels had increased significantly more in the outdoors covered samples than in the barn closed or outdoors uncovered samples (Figure 4-35). To aid interpretation, the best fit model is applied to the original May data in

Figure 4-36. This shows that the outdoors covered treatment had the lowest calcium content at the start of the experiment but the highest by the end.

Figure 4-35: Mean change in calcium content between May and November 2016 relative to the first assessment on 17th May 2016.

Error bars = 95% confidence intervals. Lettering (a,b) indicates significant differences across storage types. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.
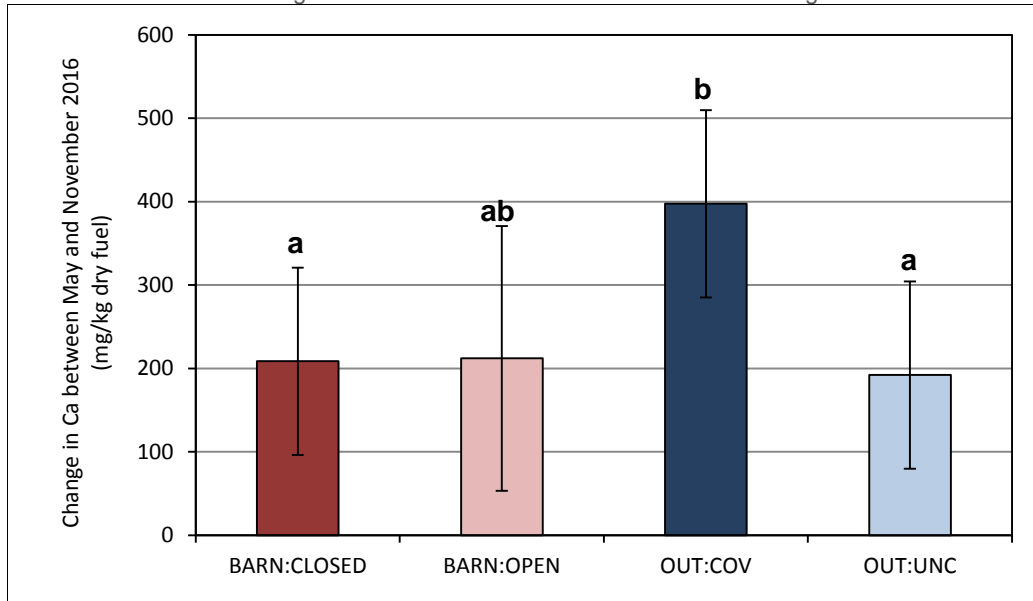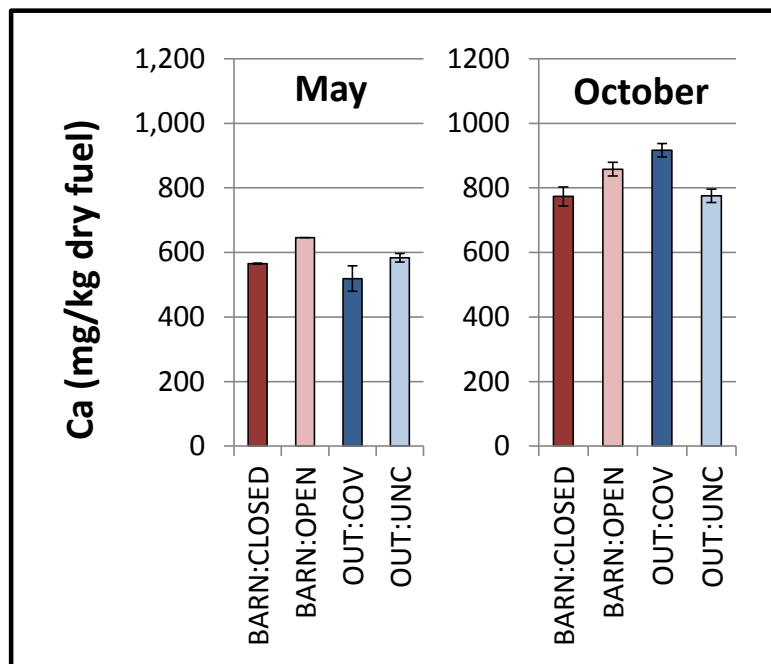

Figure 4-36: Predicted change in calcium content applied to original May 2016 data and projected through to October 2016.

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

The effects of storage regime on gross calorific value approached statistical significance ($P = 0.056$) and due to its significance for commercial operations the effects of storage type are presented in detail. Closed barn and covered outdoor samples tended to show increases in GCV from the May data, whereas open barn and uncovered outdoor samples tended to show decreases; however, these results were not statistically significant at a $P<0.05$ level (as can be seen from the large confidence intervals in Figure 4-37). To aid interpretation, the best fit model is applied to the original May data in Figure 4-38. The projected data indicate a trend for decreasing GCV with increasing exposure (i.e. from closed barn to uncovered outdoors). However, it is important to note that these projections are based on a model where estimated changes by treatment are not statistically significant based on the available data.

Figure 4-37: Mean change in GCV (kJ/kg) between May and November 2016 relative to the first assessment on 17th May 2016.

Error bars = 95% confidence intervals. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.
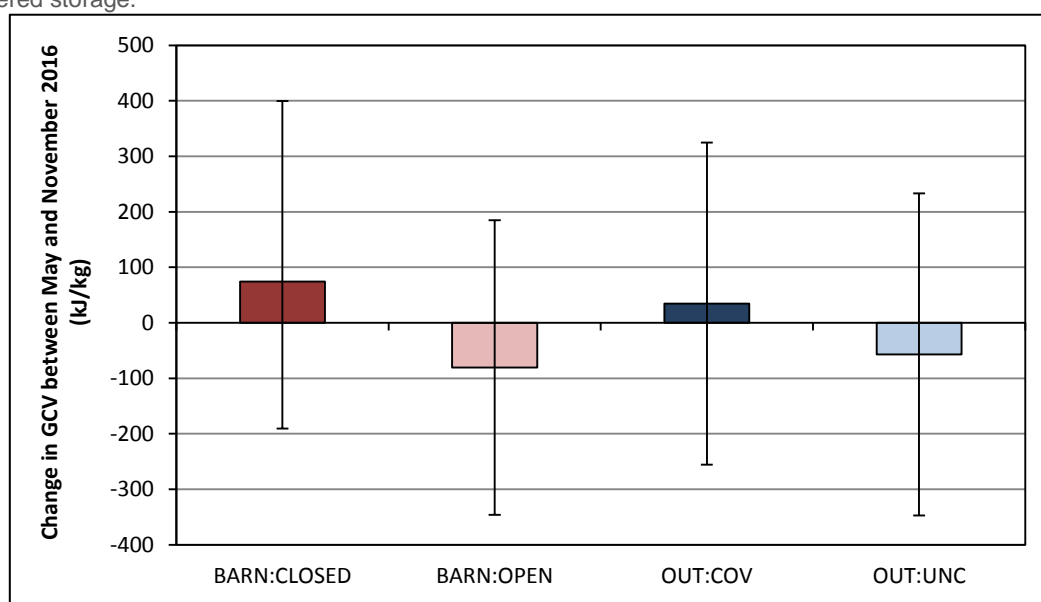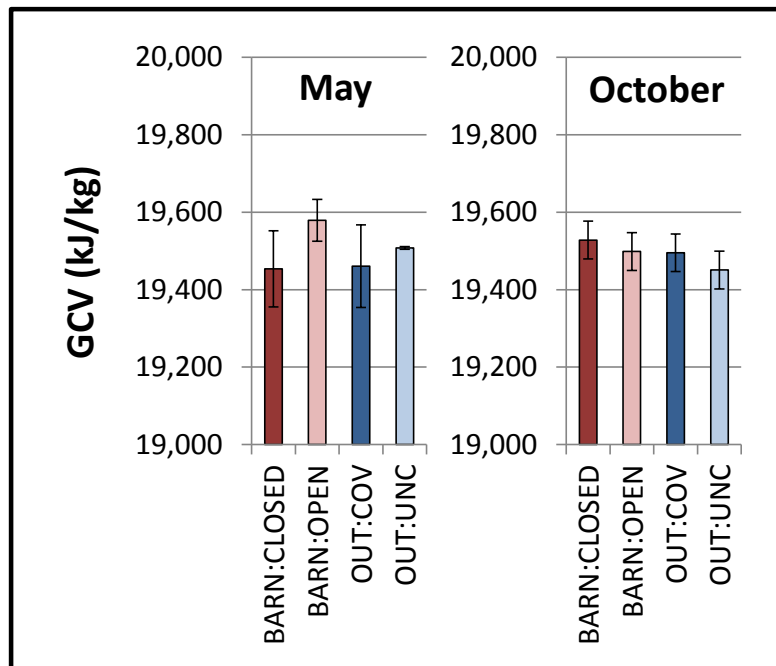
Figure 4-38: Predicted change in GCV applied to original May 2016 data and projected through to October 2016.

Error bars = +/- 1 standard error. OUT:COV = outdoor covered storage and OUT:UNCOV = outdoor uncovered storage.

4.4.3 Summary

- The experimental storage types were representative of the majority of commercial systems currently used in Britain. The 5-month storage duration represented a normal operational situation though both much shorter and longer periods may be used to fit with work patterns on the farm and market demands.
- After five months there was no difference between the composition of the bales stored in an undisturbed condition and those that had been regularly moved to allow sampling throughout the sampling period, indicating that the results on the changes through time are applicable to normal operational conditions.
- Although storage in the present experiment had no significant effect on ca 40% of the feedstock characteristics tested, several important proximate and ultimate variables were significantly influenced by the type of storage (ash, nitrogen and sulphur content); the effect on ash was also influenced by storage duration.
  - Ash levels in bales stored outdoors increased steadily whereas barn-stored bales were much higher the month after storage began and then declined so that by November the ash levels in all treatments were much the same.
  - Nitrogen content fell throughout the storage period and after five months levels were about 15% less than the initial value with the decrease being greater in the bales stored outside uncovered.
  - Sulphur increased by approximately ~40%-150% in all samples between May and November but bales stored in barns tended to have larger increases in sulphur contents than the bales stored outdoors.
- The impact of storage type on gross calorific value was very close to the threshold chosen for statistically significance (*P=0.056*). Closed barn and covered outdoor

samples tended to show increases in GCV from the May data, whereas open barn and uncovered outdoor samples tended to show decreases.

- Of the trace elements, only zinc was significantly affected by storage type; those samples stored in barns tended to show increases in zinc content (by approximately 50% over the duration of the storage) whereas the outdoors samples tended to show decreases (of approximately 20%).

- Of the halides, bromine increased during storage; unlike the other storage treatments the outdoor uncovered bales declined at first before increasing to reach similar levels to the barn stored bales by November.

- Of the ash-forming elements, calcium increased by about one third in all types of storage but especially in the outdoor uncovered bales.

- There was no effect of storage type on the metal oxides.

- Even where there was no effect of the type or duration of storage, storage did significantly increase many of the feedstock characteristics (moisture, chlorine, beryllium, vanadium, aluminium, manganese, sodium, phosphorus, silicon, the oxides of aluminium, calcium, and silicon) and decrease net calorific value, volatile matter, hydrogen, potassium, potassium oxide and the alkali index.

- Where there was a significant change during the five month's storage, in almost every instance, this indicated a deterioration of *Miscanthus* condition. The only improvements in condition were a reduction in potassium, the oxide of potassium and the alkali index.

5 Discussion

5.1 Study 5: The impact of harvest time on feedstock characteristics of *Miscanthus*

The observed changes in *Miscanthus* characteristics were supported by the literature. For example the observed decrease in N and increase in sodium in *Miscanthus* during autumn is consistent with findings of Nsanganwimana *et al.* (2016). Delaying harvesting from the autumn to the following spring improved the quality of the harvested biomass by a reduction in N, K and Cl as well as a reduction in moisture content through drying (Himken *et al.,* 1997; Heaton *et al.,* 2009*;* Di Nassi *et al.,* 2011) which is consistent with our observations on N, Cl and moisture content. In a 5-year study in Carlow, Ireland Finnan and Burke (2014) found that N mobilisation between October and February was small and most of the N decrease over this period was due to loss of leaves following abscission. This body of evidence and our own findings do not agree with Jensen *et al.* (2016) who failed to find lower levels of N, P and K in *Miscanthus x giganteus* in spring (February) than autumn (October) or a decrease in chlorine over winter.

Several of our *Miscanthus* sites, mainly those in south west England, showed a previously unreported pattern of increasing nitrogen in the late spring which may be associated with a resumption of growth in stems. In the case of nitrogen there is a view among *Miscanthus* growers in the UK (Steve Croxton, pers. comm.) that winters in some more southerly areas are not reliably cold enough to complete the growth cycle of the stems with the result that, when conditions improve in the spring, nutrients and sugars are remobilised and translocated to the overwintered stem to support a continuation of last season's growth. Although Heaton *et al.* (2009) found that there was a major reduction in N concentration between June and December but then little additional change over the following months to February/March, we have not found reports of an increase in N around March/April.

In a study that spanned 5 Degrees of latitude, contrasting soil types and land capability classes, Arundale *et al.* (2015) reported that delaying harvest from October to December decreased the proportion of hemicellulose, acetyl groups and ash but increased the cellulose and lignin at statistically significant levels. This supports our findings of a decrease in ash.

Lewandowski *et al.* (2003) reported that the reduction in yield between December and March was accompanied by a significant decrease in water content and in the concentrations of ash, nitrogen, chloride and sulphur in the biomass concentrations. Although the seasonal changes in quality we observed would generally be beneficial we did not collect yield information in either fresh or dry weight terms, therefore it is not possible to estimate the overall impact of crop quality and quantity from our project.

The nature of the changes observed reflect both active plant processes (such as cell division and photosynthesis), which will be influenced by a range of environmental cues mainly day length and temperature; passive processes such as drying, which will also be influenced by the environment, both before and after cutting; and management operations, in particular harvesting. Most of the proximate and ultimate analyses changed significantly in the course of our sampling. For any one year and site, the net effect of these is difficult to predict. Despite this the levels and trends in some *Miscanthus* characteristics, e.g. ash, NCV and calcium, were consistent from one year to another. On the other hand about half of the assessed

variables - mainly the trace elements which were generally close to the limit of detection but also several of the ash-forming elements - did not show a statistically significant pattern through time.

We anticipated that concentrations of components that are regulated by the plant would decline most markedly in the autumn and change little in the spring when the *Miscanthus* stem is essentially inert. This general pattern seems to hold for ash and calcium but also for silicon which is not mobile.

It was not possible to identify any unequivocal, simplistic correlations between either rainfall totals or intensity and changes in feedstock characteristics between the time of commercial harvesting and baling during which the cut *Miscanthus* was exposed to the weather. We considered total rainfall and average rainfall per day, whereas it is possible that the timing and distribution of rainfall events, such as the peak rainfall intensity, or heavy rain immediately after cutting, might display greater correlations. Many more samples from the period would be necessary to test rainfall impacts at greater temporal resolution.

The procedures were designed to give an insight into the feedstock characteristics of commercial *Miscanthus* over a range of potential harvesting times after the crop had ceased active growth. In two respects however the simulated harvest differed from commercial practice – operational harvesting is likely to dislodge more leaf material and break off more of the finer stalk tops, also in commercial practice the harvested crop is left on top of the stubble. The possible effects are that the chemical composition of our samples from the simulated harvested differs slightly because of the higher proportion of fine material and absence of soil contamination. There was some evidence that the levels of trace elements, e.g. molybdenum, titanium, mercury and selenium, were considerably higher after the crop had been harvested than it was in the simulated harvested in mid-March. In the case of the increase in metals in the late spring, it is more likely that the increase reflects contamination during commercial harvesting or the period when the harvested stems were lying on top of the stubble before baling. It is possible that contamination was caused by the mobile chipper used to reduce the *Miscanthus* stems to chips which could then be mixed, subsampled, and sent for chemical analysis but this would be a risk to all samples, i.e. both stems cut in a simulated harvest and stems harvested commercially, yet was not evident in the samples collected by simulated harvesting, so tends to indicate contamination is more probable from the commercial cutting system.

If the seasonal changes within *Miscanthus* are considered in the context of the five feedstocks analysed in Phase 1, it is clear that the choice of *Miscanthus* harvesting time needs to weigh up several aspects. The low moisture content of spring harvested *Miscanthus* and consequent high NCV are a clear advantage over the other feedstocks which generally had moisture contents in the range of 50-70% hence NCV's of <8000 kJ/kg as received fuel (Figs 4-3 and 4-4 respectively of D6). However it can be seen from the results that *Miscanthus* begins at this higher moisture content and lower NCV during November through January before drying down. Earlier harvesting may risk higher moisture contents than desirable. Moreover the comparison of moisture contents at the same site over two successive years suggests that the optimum harvesting time with respect to moisture content/NCV varies from year to year. There is a somewhat similar situation with ash content - although levels in *Miscanthus* at typical spring harvesting times were much less than the leaves of willow SRC and poplar SRF or the tops of poplar SRF they tended to be higher than willow SRC stems or wood of poplar SRF and spruce SRF (Fig 4-5 of D6), and the Phase 2 findings indicate that ash levels in

November ranged from 3.5 - 5% before falling to 2 - 3%. Likewise, early harvesting risks undesirably high levels of chlorine and nitrogen levels, though they too fall overwinter and are generally low. Considered as a whole these results suggest that to maximise *Miscanthus* quality, harvesting should be delayed until at least the beginning of March, with chlorine and ash a particular concern if harvesting is advanced which also risks losing the advantages of low moisture content and high NCV.

5.2   Study 6: The impact of harvest time on feedstock characteristics of willow SRC

Very few seasonal differences were detected in the willow SRC, especially when compared to the *Miscanthus* results. There are several possible reasons for the lack of seasonal differences in willow SRC. Because of the perennial nature of *Miscanthus* it was possible to compare the crop produced at a given site, however once willow SRC is harvested it is usually a further 3 years before the next harvest therefore direct comparison at a given site was not possible. Secondly there was a wider geographical range of willow SRC sites which resulted in a wider range of climate zones and soil types. Thirdly, the sampling approach was designed to take a representative sample of commercial crops – this resulted in a single *Miscanthus* genotype at all sites but several different varieties within a willow SRC site and different varietal mixes at different sites. Finally there were fewer willow SRC sampling occasions.

Although there are reports in the literature of between 40% of the N and about 60% of the P being withdrawn from willow leaves prior to abscission and stored mainly in the below ground organs, there were no seasonal trends or translocation from senescing leaves for Ca and Mg and translocation of K and S were observed only in plants grown under a low nutrient regime (Von Fricks *et al.*, 2001). It is therefore not unexpected that there was little evidence of seasonal changes in proximate and ultimate fuel properties (only GCV was affected significantly) or ash-forming elements in the present study.

By contrast, willows are well known as bio-accumulators of trace elements and heavy metals. Many experiments and trials have shown that Salix species have high rates of heavy metal uptake to the extent that they are used to remediate sites contaminated by heavy metals as reviewed by Pulford and Watson (2003) and confirmed in recent experiments by for example Tlustos *et al.* (2007) and Zarubova *et al.* (2015). For a range of willow species, concentrations of Cd and Zn in leaves, wood and bark increased towards the end of the growing season (Mertens *et al.,* 2006); likewise Migeon *et al.* (2009) reported seasonal variation in Cd, Zn, and Pb in a range of woody species including willows. Given that the soils in our studies are generally low even for rural soils, it is perhaps understandable that we observed changes only in chromium.

Our results suggest that the grower has considerable flexibility over harvesting times. If the seasonal changes within willow SRC are considered in the context of the five feedstocks analysed in Phase 1, this window should be limited to after leaf fall through to bud burst. Although inclusion of leaf material increases the GCV this risks raising the moisture content, and ash, nitrogen, sulphur and chlorine levels considerably (see Figures 4-2 to 4-8 of D6).

5.3   Study 7: The effect of variety on the feedstock characteristics of willow SRC in spring 2016

Six varieties of willow SRC (Endurance, Tora, Terra, Nova, Resolution, Sven, and Nimrod) were identified that were found at all five sites with the exception of Sven and Nimrod which were absent at Aberystwyth. Newer varieties were available at some but not all sites, and

since the main objective was to test the hypothesis that feedstock characteristics are consistent across sites we accepted older varieties that were found across a wider range of sites. At present, planting of willow SRC is very limited, being restricted on one commercial company, nevertheless Endurance, Tora, Terra, Nova, and Resolution would still be considered for any new commercial plantings.

The selection of varieties included in this study is useful in providing a comparison across varieties with a wide range of lineages. The nearest comparator – the study of willow varieties in England (Gudka, 2012) had a narrower set with only one *S. viminalis x S. Schwerinii.*

Although the sites selected for this study were research trials rather than commercial sites, the results are valuable in covering a wide geographical range and encompass a wide range of local site conditions.

The findings of the current study are consistent with many reported studies, for example. Brereton *et al.* (2014) found genotype-specific variation in all the traits they measured in an investigation of the N dynamics during growth and onset of winter dormancy in 14 willow genotypes and in a study of willow varieties in England, Gudka (2012) reported differences in cellulose, hemicellulose and lignin content, calorific value and both total ash content and the percentage of different oxides in the ash. Like Gudka (2012) this study showed that % CaO was negatively correlated with K_2O – Resolution had the lowest $CaCO_3$ while Endurance, Tora, Terra Nova and Nimrod tended to have the highest ranking whereas Resolution had the highest levels of K_2O with Endurance and Terra Nova having low levels.

One important feature of this study is that it covered a very wide range of parameters of potential relevance to the commercial operation of large-scale power plants. The results suggest a certain degree of consistency across the sites, with approximately 40% of the parameters analysed showing statistically consistent rankings. Considering the results as a whole however, two important points emerged, firstly that no variety combined the best ranking in all parameters of importance for commercial conversion technologies, and secondly for the majority of parameters, there was not a consistent ranking. Volatile matter, sulphur and chlorine content for example did not show consistent rankings and neither did the alkali index.

These results are based (quite deliberately) on a snapshot in time and the impact of seasonality should be considered. As demonstrated in Study 6, very few parameters of the mixed willow varieties showed significant seasonal trends. The plots in Appendix 5 show that most of the parameters fluctuate markedly over the three sampling times therefore the findings about the consistency of rankings should be applied to a wider time frame only with considerable caution.

As explained it is difficult to be definite about the consistency of ash rankings. In light of the importance of ash to commercial conversion plant this should be considered in any future investigations of willow SRC feedstock characteristics.

5.4   Study 8: The impact of storage after baling on *Miscanthus* composition in summer 2016

Miscanthus bales are typically densely packed but were sampled very effectively using the corer bought specifically to allow cores to be removed from positions throughout the selected bales. The bale corer had a small diameter (1.3 cm) which made collection of the cores very slow. One complete sampling of a stack took up to 4 hours, adding up to two full days each month and four days for the first and last sample collection timings. Although it would probably

have taken less time to collect the necessary weight of material using a larger core diameter, it would undoubtedly have been much harder to cut in to the bales and would probably have required a significantly more powerful drill. The smaller corer required regular sharpening due to the tough nature of the *Miscanthus* canes; a larger diameter core would need the same level of maintenance.

No significant differences were detected after five months in the condition of the bales stored in an undisturbed condition and those that had been repeatedly moved (to allow sampling). Although we had been concerned that repeated movement might make our samples unrepresentative of normal operational conditions, the results indicated that this concern was unfounded. We suggest two possible reasons: the very tight packing of *Miscanthus* within each bale effectively sealed the bale; and even with repeated sampling the stacks were opened for a relatively short time compared to the time when they were back in the stack. Regular sampling did however create one notable problem, viz. the integrity of the bales started to suffer from all the coring required to collect the agreed number of samples. If longer storage durations are investigated in the future, longer intervals between sampling times should be considered and/or each treatment should contain more bales.

The experimental storage treatments were representative of the majority of commercial systems currently used in Britain in terms of the storage method and duration. In addition to the effect on feedstock characteristics discussed below, it was noted that there was a potential Health and Safety risk when workers were installing the tarpaulin covering over the top of the stack, over the stack edges and down the sides; in windy conditions the edges were not always obvious when the wind billowed up below the tarpaulin.

This study reports for the first time that more than half of the tested variables, including many important practical feedstock characteristics, were changed significantly during storage. The majority of these changes decreased fuel quality. From a practical point of view, some period of on-farm storage is likely to be needed so the question becomes: what can be done to minimise the deterioration? Advice from Terravesta is that "ideally the best form of storage for your *Miscanthus* is an indoor site with a dry floor" (pers. comm. Mos, 2015) but in fact our study indicated that sulphur and zinc increased more in both types of indoor storage compared to outdoor storage. Furthermore ash values were much higher in the covered storage in the samples after one month's storage. Nitrogen decreased (improved) more in the outdoor uncovered bales but this type of storage also increased calcium more than the other treatments. These results suggest that no single type of storage is likely to minimise the deterioration in all aspects of feedstock quality and the choice of storage type is more likely to be dictated by what is available on the farm, especially in the absence of any price differential linked to quality.

Of the provenance information, rainfall in spring 2016 was considered in some detail. During the period of the sampling for Study 8, rainfall was relatively low compared to typical values. In April and July rainfall in the Taunton area was 50-75% of the 30-year average (over 1981-2010). The *Miscanthus* used for the storage experiment lay in the field for 18 days prior to baling on 5th May therefore the low April rainfall is consistent with the very low moisture content of the bales (~8 wt %) when they were stacked. Rainfall in May, August and September was close (75-125%) to the average, while June was considerably wetter (125-150% of the 30-year average). Despite this monthly deviation from the average, rainfall in South West England and South Wales was slightly below average in both spring (96% of the average) and summer (99%) 2016, and significantly below average (78%) in the autumn.

For those bales of *Miscanthus* stored outside, especially uncovered, the level of rainfall may be expected to influence changes of some parameters during storage. This applies most obviously to moisture content but rainfall may also have other effects such as leaching of water soluble minerals or contaminating bales with rain borne atmospheric pollutants. The extent to which these effects are observed may be expected to correlate with rainfall levels, especially for those stacks in the more exposed storage methods. In fact as discussed below there was not an obvious impact of rainfall on feedstock quality during storage.

Although there appears to be some slight variation and fluctuation between the different storage methods, moisture content increased, which was reflected in a reduction in net calorific value, even in the treatments where the bales had protection. Furthermore there was no significant difference between storage treatments. This was unexpected in several ways. We anticipated that moisture content would be maintained or even reduced during storage in the bales stored indoors and that in the bales stored outside, moisture content would possibly rise and also be more variable in the uncovered bales than those that were covered over the top and down most of the sides of the stack. Even if we consider moisture contents following the wet month of June 2016, the overall increase appeared not to be significantly higher in the outdoor stored samples than those stored in a barn, suggesting that rainfall might not be the primary cause of the increase. It is possible that the generally humid conditions at the site meant that even bales stored inside absorbed moisture. In fact, there was a high rainfall event at the experimental site less than two weeks after bales entered storage which may have contributed to prolonged periods with high atmospheric humidity. On the other hand this makes it difficult to understand why the moisture content of the outdoor uncovered bales was not higher or more variable than the other treatments – possibly the outdoor uncovered bales were better ventilated. Normally chlorine content is inversely related to moisture content yet during storage both have increased.

Sulphur increased more during inside than outdoor storage though the absolute levels were so low that differences would not be of operational significance. One possible explanation is that the greater ventilation on the outdoor bales may have limited the increase in sulphur. Better ventilation of the outdoor uncovered bales may also explain the greater reduction in nitrogen compared to both the outdoor covered and indoor bales. Although the nitrogen dynamics are interesting, the levels are so low that they would not be a concern to an end user; also the variability falls within typical analytical ranges of sample variation.

We cannot offer an explanation for the general increase in sulphur or the storage-related differences in zinc or bromine.

Although the changes observed in gross calorific value would be within an acceptable tolerance of the sample process for *Miscanthus,* there was a trend for GCV to increase in closed barn and covered outdoor samples but decrease in open barn and uncovered outdoor samples tended. One suggestion was that leaf material was blown away in the more exposed conditions, however given the tight packing of both bales and stacks this is not a convincing suggestion, thus there is no obvious reason for this finding.

Contamination by wind-blown lime or soil may explain the general increase in calcium levels. The soil in the yard and surrounding fields has a high calcium content (>80% pers. comm. R. Gothard; farmer at the site). The increase was especially marked in the outdoor covered bales - perhaps the cover trapped more fine particulates including those with a high calcium content.

One particularly surprising aspect of the results was the increase in the content of ash, and many of the metals including aluminium and silicon, regardless of storage treatment. Possibly this was the result of contamination, either from wind-blown soil particles, soil introduced during stacking of the bales, or during the sampling process. The pattern of ash levels (
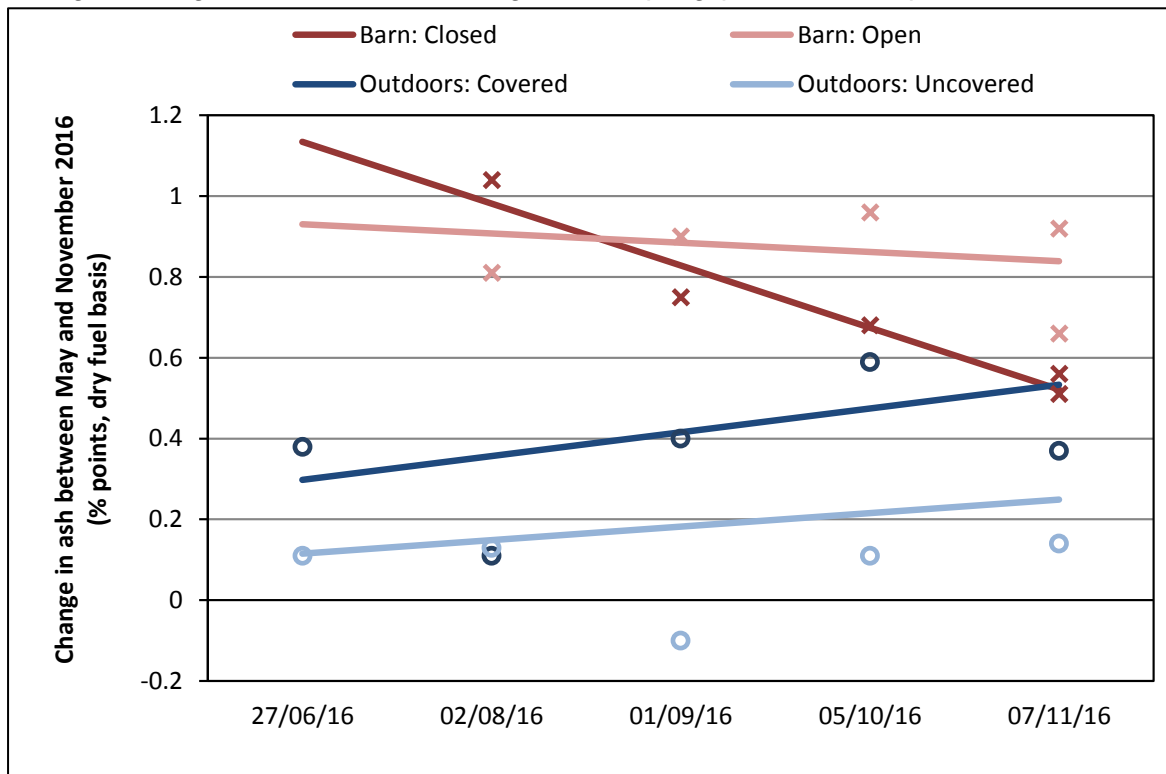

Figure 4-26) hints that much of the increase is due to dust being stirred up when the bales are being stacked at the start of the storage period; this may also explain why the early increase was worst in the closed barn which could have been dustier. All of these effects may be exacerbated at this site since the soil in the neighbourhood had a high calcium concentration.

In contrast to many of the changes discussed above, potassium levels were significantly lower after storage than before; this translated into a significant decrease in alkali index to a level where fouling is unlikely. While the reduction in potassium in outdoor bales could be a result of leaching, this is unlikely to explain the changes in barn-stored bales.

The changes in feedstock quality provide extremely interesting and commercially relevant findings but should be used with caution bearing in mind that they relate to just one site and year. In addition, the storage treatments used elsewhere may differ in small but important ways even though they would be given the same general description as ones used here. For example, outdoor covered storage may use different types of tarpaulin or cover more/less of the stack sides.

Although the 5-month storage duration represented a typical operational situation, the questionnaire showed that both much shorter and longer periods may be used to fit with work patterns on the farm and market demands. Since this project demonstrated major changes in many aspects of *Miscanthus* quality during storage and also that the storage method and duration could be influential, these findings should be considered carefully by the sector.

6   Conclusions

Many of the *Miscanthus* characteristics of crops grown at six sites ranging from Lincoln to south west England changed significantly through time. The majority of values decreased over the sampling period – early November 2015 to late May 2016 – for example moisture content, gross calorific value, carbon, chlorine, barium, zinc, bromine, calcium, phosphorus, and silicon. Of these, many showed a decreasing trend throughout the period during the time both as a standing crop (November 2015 to March 2016) and also as the crop was harvested commercially and left on the stubble until baling (until late May at the longest). There were however interesting variants of this general decreasing trend. A smaller number of variables increased between November 2015 and June 2016 – for example Net Calorific Value, volatile matter, K_2O - and in one case (sodium) levels apparently increased through the winter and early spring but then decreased again.

If the seasonal changes within *Miscanthus* are considered in the context of the five feedstocks analysed in Phase 1, it is clear that the choice of *Miscanthus* harvesting time needs to weigh up several aspects. Considered as a whole, our results suggest that to maximise *Miscanthus* quality, harvesting should be delayed until at least the beginning of March, with chlorine and ash a particular concern if harvesting is advanced which also risks losing the advantages of low moisture content and high NCV. This study therefore supports the generalisation put forward by Heaton *et al. (*2009): there is a trade-off to consider when harvesting perennial biomass crops: harvest too late and yield declines, harvest too early and risk higher mineral contents, particularly nitrogen.

The findings were consistent with the literature and confirm a general decrease through late autumn, winter and early spring in moisture content, ash, carbon, nitrogen, chlorine, molybdenum, zinc, bromine, phosphorus, silicon, and calcium accompanied by an increase over the same period in net calorific value, volatile matter, and sodium. Several sites, mainly those in south west England, showed a previously unreported pattern for *Miscanthus* of increasing nitrogen in the late spring (2016) which may be associated with a resumption of growth in stems.

We investigated the effect of total rainfall and average rainfall per day and changes in parameters between cutting and baling the *Miscanthus* crop. It was not possible to draw any firm conclusions about the impact of rainfall.

A comparison between the same calendar date in 2015 and 2016 suggested that the *Miscanthus* was drier in 2015, with the result that for a given site some characteristics, e.g. chlorine, were lower while calcium was higher date for date in 2015. With these exceptions the levels were generally similar in the two years up to the point of harvesting and the trends were similar in the two years. Changes between harvesting and the pre-baling sample were less consistent across sites and years.

There was some evidence that the levels of trace elements were considerably higher after the crop had been harvested than it was in the simulated harvested in mid-March which probably reflects contamination during commercial harvesting or the period when the harvested stems were lying on top of the stubble before baling.

Very few seasonal differences were detected in the willow SRC, especially in relation to the *Miscanthus* results. While there are several possible reasons for the lack of seasonal

differences in willow SRC, the results are not unexpected. There is very limited literature about seasonal changes in proximate and ultimate fuel properties or ash-forming elements (only the change in GCV was significant in the present study) and although willows are well known as bio-accumulators of trace elements and heavy metals, the soils at the sites studied were very low in both.

Our results suggest that willow SRC growers have considerable flexibility over harvesting times. This window should be limited to after leaf fall through to bud burst because inclusion of leaf material risks raising the moisture content, and ash, nitrogen, sulphur and chlorine levels considerably.

There was a certain degree of consistency in willow variety properties across sites from Northern Ireland to Southern England, with approximately 40% of the parameters analysed showing statistically consistent rankings for the varieties tested (Endurance, Nimrod, Resolution, Sven, Terra Nova, and Tora). The most highly significant differences ($P<0.01$) in rankings were in moisture, net calorific value, carbon, nitrogen and $CaCO_3$. For example, Endurance was consistently the lowest in terms of moisture content, with Tora, Resolution and Sven in the mid-range, with Terra Nova with Nimrod generally having the highest moisture content. Resolution generally had low nitrogen concentrations whereas Nimrod, Terra Nova and Endurance generally had high nitrogen levels with Sven intermediate. In addition, differences in the ranking of gross calorific value, copper, calcium, magnesium, manganese, K_2O, MgO and Na_2O were significant with $P<0.05$ and >0.01. For example, Nimrod had consistently high gross calorific value, Tora had consistently low levels of copper, while Resolution, followed by Sven, had consistently low levels of calcium.

Considering the results as a whole, there was evidence of consistent differences across a wide geographical range in approximately 40% of the important parameters analysed but no variety combined the best ranking in all parameters. Conversely our results suggest that for the majority of parameters, there was not a consistent ranking. Volatile matter, sulphur and chlorine content for example did not show consistent rankings and neither did the alkali index.

These results are based (quite deliberately) on a snapshot in time and the impact of seasonality should be considered. Study 6 showed that most of the parameters fluctuated markedly over the three sampling times therefore the findings about the consistency of rankings should be applied to a wider time frame only with considerable caution.

The experimental storage treatments were representative of the majority of commercial systems currently used in Britain in terms of the storage method and duration. Although storage in the present experiment had no significant effect on ca 40% of the feedstock characteristics tested, there was a significant change in the other parameters during the five months' storage, and in almost every instance this indicated a deterioration of *Miscanthus* condition. Although the method of storage did not affect levels of potassium, the oxide of potassium or the alkali index, these important characteristics were all significantly lower after storage, which represented improvements in condition.

From a practical point of view, some period of on-farm storage is likely to be needed so the question becomes: what can be done to minimise the deterioration? Although industry advice is that indoor storage with a dry floor is preferred, our study indicated that sulphur and zinc levels increased more during indoor storage compared to outdoor storage. Furthermore ash values were much higher in the covered storage in the samples after one month's storage. Nitrogen decreased (improved) more in the outdoor uncovered bales but this type of storage

also increased calcium more than the other treatments. Moisture content increased, even in the treatments where the bales had protection, and there was no significant difference between storage methods; although these findings indicate that covered storage may not be necessary even in high-rainfall areas these findings must be validated at other sites. Taken as a whole these results suggest that no single type of storage is likely to minimise the deterioration in all aspects of feedstock quality and the choice of storage type is more likely to be dictated by what type of storage is available and perhaps the contamination risk on the farm, especially in the absence of any price differential linked to quality.

Although the 5-month storage duration represented a typical operational situation, the questionnaire showed that both much shorter and longer periods may be used to fit with work patterns on the farm and market demands. Since this project demonstrated major changes in many aspects of *Miscanthus* quality during storage and also that the storage method and duration could be influential, these findings should be considered carefully by the sector.

7 Key findings

- The sampling procedures were robust and consistent giving a high degree of confidence in the dataset.

- Many of the *Miscanthus* characteristics of crops grown at six sites ranging from Lincoln to south west England changed significantly through time.

- In *Miscanthus* a general decrease through late autumn, winter and early spring was observed in moisture content, ash, carbon, nitrogen, chlorine, molybdenum, zinc, bromine, phosphorus, silicon, and calcium accompanied by an increase over the same period in net calorific value, volatile matter, and sodium.

- It was not possible to draw any firm conclusions about the impact of rainfall on changes in parameters between cutting and baling the *Miscanthus* crop.

- Considered as a whole these results suggest that to maximise *Miscanthus* quality, harvesting should be delayed until at least the beginning of March, with chlorine and ash a particular concern if harvesting is brought forward which also risks losing the advantages of low moisture content and higher NCV.

- Several sites, mainly those in SW England, showed a previously unreported pattern of increasing nitrogen in the late spring 2016 which may be associated with a resumption of growth in stems following mild winter conditions.

- Only a few characteristics of willow SRC grown at six sites from north west to southern England showed statistically significant differences across three simulated harvesting times (mid-November, mid-January and mid-March) – GCV (DAF), chromium (d), $CaCO_3$ (na), K_2O (na), and P_2O_5 (na) - with the majority showing no difference. For the characteristics that did change, a variety of patterns was evident.

- Gross calorific values of willow SRC at all sites fell between mid-November and mid-January; by mid-March the GCV was either similar or had increased. Data from spring 2015 suggested that the increase in GCV may continue through to at least early May.

- There are several possible reasons for the lack of seasonal differences in willow SRC, especially in relation to *Miscanthus*, but the results are not unexpected. There is very limited literature to support seasonal changes in proximate and ultimate fuel properties or ash-forming elements (only GCV was significant in the present study) and although willows are well known as bio-accumulators of trace elements and heavy metals, the soils at sites studied were very low in concentrations of both.

- Our results suggest that willow SRC growers have considerable flexibility over harvesting times. This window should be limited to after leaf fall through to bud burst because inclusion of leaf material risks raising the moisture content, and ash, nitrogen, sulphur and chlorine levels considerably.

- There was a certain degree of consistency in willow variety properties across sites from Northern Ireland to Southern England, with approximately 40% of the parameters analysed showing statistically consistent rankings for the varieties tested (Endurance, Nimrod, Resolution, Sven, Terra Nova, and Tora). Considering the results as a whole no variety combined the best ranking in all parameters.

- For the majority of parameters however, there was not a consistent ranking. Volatile matter, sulphur and chlorine content for example did not show consistent rankings and neither did the alkali index.

- The experimental storage treatments were representative of the majority of commercial systems currently used in Britain in terms of the storage method and duration.
- A core sampler driven by a hand-held drill allowed representative samples to be collected from within *Miscanthus* bales.
- This study makes available for the first time evidence on the impact of storage type and duration on important feedstock characteristics. The findings can be grouped into three:
  - ca 43% of the feedstock characteristics tested were not significantly affected by storage
  - another 43% were affected by storage but there was no influence of storage treatment; the majority of these changes decreased fuel quality.
  - in the remaining 14% of analysed feedstock characteristics, which included ash, nitrogen, sulphur, zinc, bromine and calcium, storage treatments did have a significant influence; again the majority of these changes decreased fuel quality.
- These results suggest that no single type of storage is likely to minimise the deterioration in all aspects of feedstock quality and the choice of storage type is more likely to be dictated by what type of storage is available and perhaps the contamination risk on the farm, especially in the absence of any price differential linked to quality.

8 Acknowledgements

9 References

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10 Appendices

1. Table of removed outliers
2. Graphs of all *Miscanthus* feedstock characteristics at the five sampling times
3. Graphs of comparable *Miscanthus* feedstock characteristics in Spring 2015 and 2016
   Note that data points from 2015 are overlaid on the equivalent Julian day of 2016 with the x axis showing only the Phase 2 labels
4. Graphs of all willow SRC feedstock characteristics at the five sampling times
5. Graphs of comparable willow SRC feedstock characteristics in Spring 2015 and 2016.
   Note that data points from 2015 are overlaid on the equivalent Julian day of 2016 with the x axis showing only the Phase 2 labels
6. Graphs of all willow SRC feedstock characteristics for six varieties (Endurance, Nimrod, Resolution, Sven, Terra Nova, and Tora) at four-five sites (Aberystwyth, Brook Hall, Long Ashton, Loughall, and Rothamsted)
7. Statistical analysis of willow SRC varietal rankings by feedstock characteristic
8. Questionnaire on *Miscanthus* storage
9. Experimental protocol on *Miscanthus* storage
10. Summary of questionnaire responses on *Miscanthus* storage