Published version: https://doi.org/10.1016/j.biombioe.2016.06.027; Licence: CC BY-NC-ND

Accepted version:

Influence of the agricultural management practices on the yield and quality of poplar biomass (a 9-year study)

M. J. Fernández*, R. Barro, J. Pérez, J. Losada and P. Ciria Centro de Investigaciones Energéticas Medioambientales y Tecnológicas (CIEMAT) CEDER-CIEMAT (Energy Department, Biomass Unit), Autovía A-15, sal. 56 - 42290 Lubia, SORIA (Spain) *Corresponding author, Tel: +34 975 281013, Fax: +34 975 281056, E-mail:

miguel.fernandez@ciemat.es

ABSTRACT: The aim of this work is to study the effect of different agricultural management on the yield and quality of two poplar biomass clones (AF2 and I-214 clones) in short rotation coppices (SRC), which were harvested using different alternatives (with and without cutting and sprouting after the first year), with two fertilisation doses and through three different 3year rotation cycles. The plantation was established in 2006 in a marginal land at 1,100 m above sea level in central-northern Spain. Yields were evaluated and biomass samples were analysed to determine the quality of the biomass for energy purposes. Biomass quality was estimated taking into account calorific value, volatile matter, ash content, carbon, nitrogen, sulphur and chlorine contents, as well as the chemical composition and melting behaviour of their ashes.

The highest yields, around 9 dry tons per hectare and year, were obtained in this marginal land during the first and second rotation cycles when plots received a supplementary fertilisation. Both clones (AF2 and I-214) provided similar yield and biomass quality. Plots where poplar was not harvested the first year (without cutting and sprouting after the first year) provided higher accumulated yields. Poplar biomass from SRC can be considered a suitable solid biofuel due to its appropriate ash melting behaviour and its low content of nitrogen (0.44 wt-%), sulphur (0.03 wt-%) and chlorine (around 0.01 wt-%). No important significance effect on the poplar quality can be found depending on the additional fertilisation. Poplar quality varied as a function of root/stem age.

Keywords: biomass; poplar; yield; quality; composition; combustion.

1 INTRODUCTION

The biomass demand from non-food dedicated crops and agro-industrial residues is expected to increase in Europe due to the progressive development of the bioenergy and the bioeconomy [1-2]. The available agricultural surface could be partially dedicated to produce biomass for bioenergy and, in particular, the settlement of dedicated energy crops could offer new opportunities to current traditional agriculture in less-productive/non profitable agricultural areas.

Poplar is currently one of the most studied species worldwide as a potential source of solid biomass for combustion to generate heat and electricity. Some of the advantages reported of poplar in short rotation coppice (SRC) are: its high biomass and energy yields, its ecological interest and its comparatively low biomass production cost [3-5]. Nevertheless, relatively little information is available in the literature on the behaviour of the crop in SRC conditions in adverse pedoclimatic conditions, as those of many low productive or abandoned agricultural lands in southern EU countries.

The optimal selection of poplar clones, fertilisation rates and the harvest moment are crucial parameters to reduce the overall costs of the crop, not only the ones derived from biomass production, but also those associated with the combustion process, since the cited parameters could affect the biomass quality as a fuel [4-8], playing an important role in the appearance of slagging, fouling, corrosion or emission problems. Therefore, the crop management practices should be optimised and selected not only in terms of obtaining the highest biomass yields, but also taking into account the biomass quality properties in order to increase the efficiency of the thermal conversion plants and minimise the environmental impact. The literature is very scarce regarding the composition and quality of poplar biomass SRC as a function of crop management practises.

The international multipart standard ISO 17225 has recently set the fuel specifications and classes for particular trade forms of biomass, including pellets [9], briquettes [10] and wood chips [11], among others. The quality requirements regarding properties such as ash, nitrogen, chlorine, sulphur or trace elements are clearly specified. These properties must be determined, particularly if biomass is used in small-scale applications, as some woody energy crops might exceed the standarised limits.

In the above context, this study offers valuable information about the yields and quality of the biomass obtained from two selected poplar clones grown under SRC in low productive agricultural areas with stringent pedoclimatic conditions. The effects that the number of harvests and the inorganic fertilisation dose have on the yield and fuel quality of the biomass obtained have also been determined for two poplar clones along three rotation cycles (9 years). The offered results are essential in the planning and decision-making processes for the eventual future commercial implementation of the studied energy crop as an alternative in low productive agricultural areas.

2 MATERIALS AND METHODS

2.1 Plantation establishment

The plantation was set in March 2006 using two poplar clones: I-214 and AF2 (*P. x canadensis* Moench *P. deltoides* March *x P. nigra* L.). I-214 is the traditional clone utilised in the plywood industry in Spain due to the high yields it provides. It is used in this study as a reference clon because of its widespread cultivation in Mediterranean areas [12-13]. AF2 was specifically selected in Italy [14-15] for biomass production in short rotation conditions. It is a very productive clone, providing stable yields at a wide range of sites and under different coppicing managements [14].

A total of 800 unrooted 25-cm long poplar cuttings were planted manually at a density of 33,333 plants/ha (1 m x 0.30 m spacing). An experimental trial with a split-plot design with

four replications was used. Each replicate subplot consisted in 25 cuttings of the same genotype, resulting in square experimental units in which the 9 central cuttings were monitored in order to avoid the border effect (Figure 1).

The SRC poplar plantation was located in the Spanish province of Soria. The plantation site is situated at 41°36′ N and 2°30′ W, at an altitude of about 1,100 m above sea level. The climatic conditions are Mediterranean continental, with a free frost period only between May and September. In general, winter months in the area are cold, with mean temperatures around 3-4 °C, minimum temperatures falling below 0 °C, frequent frosts (89 days per year) and occasional snows (25 snowing days per year). Summers at this site are fairly hot, with mean temperatures around 20 °C and maximum temperatures well above 30 °C (see Table 1). Annual pluviometry averages about 500 mm. It is important to note that irrigation was applied according to crop requirements and weather conditions. Plots were drip irrigated during the dry months (from May to September). Altogether, it could be considered the plantation area is established in a marginal climatic zone for the cultivation of poplar.

The soil at the experimental site had a sandy texture, with 86% sand, slit and clay less than 10%, low organic matter (0.6%), and low contents of available P (6.6 ppm) and K (61 ppm). This soil was considered poor, light and with good drainage.

To estimate biomass yields, 36 trees per condition (9 trees per replication) were cut at the end of 2008, 2011 and 2014 (3 rotation cycles). Plants were severed at the ground line (10 cm above the soil level) after litterfall, and the weight of the aerial biomass (stems and branches) was recorded. Trees were cut following (Figure 2) two different harvesting schemes (A and B). In half of the plots (harvesting alternative A), nine trees were additionally cut at the end of 2006, when they were 1-year old, to evaluate whether this additional cut would boost crop productivity during the following years.

Table 2 can be also consulted for a better comprehension of the codes used. The first letter and number indicates the age of the root in years, whereas the second part of the code indicates the age of the stem. In alternative A, poplar trees were cut at the end of the first year (biomass R1S1: trees with a 1-year old root and a 1-year old stem). This same plantation was cut again two years after sprouting (biomass R3S2: trees with a 3-year old root and a 2-year old stem). In contrast, trees of alternative B were cut only once in the first rotation cycle (3-year old). Two more 3-year rotation cycles were evaluated in both plantations (A and B).

A scheme of the applied fertilisation as a function of the harvest alternative and root/stem age is shown in Table III. Before planting and additionally in 2007 and 2009, NPK fertilisation (8-15-15) was applied at a dose of 600 kg/ha (basic fertilisation, BF). Each year (2006, 2007, 2009 and 2013), half of the trees in all plots received additional fertilisation (AF) with NPK (12-12-17), elements (6% S and 1.2% Mg), and microelements (0.1% Fe, 0.02% B and 0.01% Zn), at 800 kg/ha. The amount of nutrients applied as base fertilisation (BF) was calculated taking into account the extractions expected by the crop according to our own previous studies [16]. As the soil where the plantation was established can be considered a poor soil, additional fertilisation (AF) was applied to half the plot in order not to limit the potential biomass production. Taking into account sustainability criteria, N, P, and K applications decreased as trees aged (Table 3) because these nutrients are supposed to be partially supplied when poplar leaves fall and decompose [17].

Deltamethryn 2.5 w/v-% was applied at the beginning of each vegetative period for beetle

(Melasoma populi) control.

2.2 Sampling and analytical methods

Biomass quality was determined by analysing three whole trees (stem and branches) per experimental condition. Every tree was selected randomly, and was completely chipped and milled. After grinding, homogenisation, dividing and drying, a representative analytical biomass sample was obtained, following the European standard of sample preparation EN 14780. Analytical tests were performed in the Laboratory of Biomass Characterisation of CEDER-CIEMAT.

The determination of moisture, ash, volatile matter, chlorine (Cl), sulphur (S), carbon (C), hydrogen (H), nitrogen (N) and calorific value were carried out following European standards for Solid Biofuels developed by the Technical Committee CEN/TC 335. Moisture content was determined by drying the biomass in an oven at 105 °C to constant weight, following the European standard EN 14774-2. Volatile matter and ash content were determined following EN 15148 and EN 14775 standards, respectively. Calorific value was determined using an automatic calorimeter IKA C-5000 by applying EN 14918. C, H and N were determined according to EN 15104 by using a TruSpec (Leco) elemental analyser equipped with infrared detectors and a thermal conductivity detector. The determination of Cl and S was carried out by ion chromatography after sample combustion in a calorimetric bomb and the ulterior recovery of chloride and sulphate in an aqueous solution (EN 15289).

Ash samples, which were obtained at 550 °C, were digested in a microwave furnace using HNO₃, H₂O₂ and HF in a first step and H₃BO₃ in a second step according to EN 15290, and inorganic elements including aluminium (Al), calcium (Ca), iron (Fe), magnesium (Mg), manganese (Mn), phosphorous (P), potassium (K), silicon (Si), sodium (Na), titanium (Ti) and zinc (Zn) were analysed by ICP-AES using a Thermo Jarrell Ash simultaneous spectrometer. Arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), nickel (Ni) and lead (Pb) were analysed according to the European Standard EN 15297 by ICP-MS (iCAP Q, Thermo Fisher Scientific). Mercury (Hg) was determined by thermal decomposition, gold amalgamation and atomic absorption spectrophotometry using a direct mercury analyser (DMA-80, Milestone).

Ash fusibility was based on the changes in shape detected during the heating of a cylindrical ash pellet (ash produced at 550 °C) from room temperature to 1,400 °C in air atmosphere. The characteristic temperatures measured by an optical heating microscope were: deformation, hemisphere and fluid temperatures, following the CEN/TS 15370-1 pre-standard.

2.3 Statistical analyses

Data were analysed by statistical software (Statgraphics Plus 5.0) in order to check the statistical significance of the differences between clones, additional fertilisation and root/stem ages on the production and the quality parameters. The Statgraphics software was used for performing typical analyses of variance (ANOVA). Significant effects were revealed at = 0.01. The Fisher's least significant difference (LSD) at 95.0% probability was used to

evaluate the differences among mean results.

3 RESULTS AND DISCUSSION

3.1 Biomass yields

The averaged mass recorded per tree, together with the standard deviations and the number of trees evaluated per condition, are shown in Table 4. The survival rate was higher than 89% in all harvests, reaching 100% in some of them. This means that between 32 and 36 trees were measured per condition.

Both clones (AF2 and I-214) provided similar biomass yields without statistical significance differences. Therefore, it seems that they are both similarly adapted to the harsh and unfavourable edaphoclimatic characteristics at the site of the plantation. Biomass yields are shown in Figure 3 as a function of the age of stems and roots for both fertilisation doses and considering all clone data. Plots receiving a supplementary fertilisation provided significantly higher yields. The first and second rotation cycles provided the highest partial yields, which averaged about 9 dry tons per hectare and year.

Regardless of the fertilisation applied, cutting the trees when they were 1-year old did not boost yields. As shown in Table 5, the plots where biomass was not harvested the first year (harvest alternative B) provided higher accumulated yields, 69 Mg ha⁻¹, as opposed to 58 Mg ha⁻¹. The harvesting alternative B provided 28 Mg ha⁻¹ for the first and second rotation cycles and 13 Mg ha⁻¹ during the third rotation cycle. Plots where trees were cut when they were 1-year old (harvest alternative A) provided lower accumulated yields, 24, 24 and 10 Mg ha⁻¹ during the first, second and third rotation cycles, respectively. Therefore, from a point of view of biomass production and also from an economic perspective (the overall costs of performing an additional cut), harvesting scheme B is the preferred alternative under the conditions studied.

Combining the yields obtained for harvesting alternatives A and B, the effect of the additional fertilisation was estimated to be about 2 times higher than the accumulated yield when only basic fertilisation was applied. Biomass production dropped off dramatically with the number of rotation cycles (Table 5). Biomass drop in the third rotation cycle could indicate the end of the poplar productive cycle.

3.2 Biomass characteristics and biomass quality

In this section, the physical and chemical characteristics of the I-214 and AF2 biomass obtained from 3 rotation cycles are presented and the influence of the growing conditions (fertilisation and harvesting scheme) on the fuel characteristics are evaluated. In addition, the combustion qualities of the pellets and chips that could be produced from this biomass are discussed and compared to the requirements of the current international standards.

3.2.1 Physical and chemical characteristics

Table 6 shows the calorific values, as well as the proximate and ultimate analyses for the obtained poplar biomass.

Water content is a highly relevant quality parameter due to its impact on the efficiency of the energy transformation, transport and storage. Moisture content of poplars at harvest time was typically very high (50-60%) and thus, it has to be reduced. Frequently, the biomass storage period is used for drying purposes and so the best possible storage practices and conditions must be used in order to assist the drying process. Degradation and significant losses of the material, mould development, or changes in the overall quality of the fuel should be avoided. Previous tests at the experimental site demonstrated that the drying process when poplar is stored outdoors is slow [18]. Moisture levels of 30% can be naturally reached at the beginning of the spring. However, once stems are dried, their own bark layer will be able, at least up to some extent, to prevent water from penetrating in the wood as easily as it does when chips are left in the field. Therefore, a low water level in poplar stems could potentially be maintained for longer periods.

Generally, woody biomass has lower ash content than herbaceous biomass. When it is entirely debarked, its ash content is usually very low (<1 wt-%) as it can be seen in the standard ISO 17225-1, Table 1 [19]. It is worth considering that the ash content can increase considerably if stones, sand and/or dust are adhered to the biomass during cutting, collection or handling. In this study, the pollution of the biomass during collection was prevented from happening as poplar biomass was sampled manually. This fact may also explain the very low contents of Si, Al, Fe and Ti (see Table 9) observed, which are typical elemental soil constituents. The average ash content of the poplar SRC biomass in this study was 2.7% on dry basis, 5-9 fold higher than the ash content of debarked pine sawdust, which is used as a reference of high-quality biomass. According to the international standards 17225-2 [9] and 17225-4 [11], pellets and chips from the considered poplar should be included in industrial pellets (class I3) and in domestic chips (class B), respectively.

Poplar S and Cl contents, elements related to acid rain, corrosion, and sintering problems [20-21] were low: 0.03% and 0.01%, respectively, and thus the strict limits set by international standards for the best quality wood pellets (class A1) can be fulfilled [9]. However, the class limitation sets by ISO 17225-1, Table 1 [19], and ISO 17225-2 [9] standards, classifies the biomass obtained from this short rotation coppice as A2 or B classes, due to their origin/source. Regarding environmental contamination from poplar combustion, it can be determined that the degree of pollution could be negligible by considering not only the low content of sulphur and chlorine in poplar but also an important proportion of sulphur and chlorine, are maintained in the ashes deposited on cool surfaces of any thermal plant [23]. By comparing poplar (0.03% S) with other fuels, sulphur emissions by chimney are lower than in some fossil fuels, e. g. gas-oil for domestic applications (0.1% S), according to the Directive 1999/32/EC relating to a reduction in the sulphur content of certain liquid fuels.

Like SO₂, NOx also contributes to acid rain and the formation of harmful particulate matter [21]. In woody biomass, N content is usually lower than 0.5% (aerial biomass). In contrast, woody biomass from pruning trees and shrubs can easily exceed this value, as N is concentrated mainly in leaves, and also in twigs, although to a less extent. As N averaged 0.44% (see Table 6), the biomass obtained from this study falls into the A2 class, according to the international 17225-2 standard, which sets a maximum value of 0.3% for the A1 class [9]. Contrary to forecasts of SO₂ emissions, NOx emission predictions are more difficult. The

formation of fuel NOx is due to the oxidation of nitrogen in the fuel and it is of considerable significance during the combustion process.

Net calorific values around 19 MJ kg⁻¹ in dry basis are usually typical calorific values of woody biomass such as forest residues and residues derived from their associated industries. Mean value of poplar was 18.25 MJ kg⁻¹ db (see Table 6) and it seems to be a little lower than those of 19 MJ kg⁻¹ db. This variation could generate not to fulfil the limits of the new ISO pellet standard (16.5 MJ kg^{-1} as received, see reference [9]). In order to fulfil this limit and as the most important variation of the calorific value is due to its strong dependence with the moisture, a dry should be carried out until values lower than 9% wb, instead of 10% as is shown in the ISO pellet standard.

A multifactor analysis of variance (ANOVA) was carried out, taking into account the following three factors: fertilisation dose, clone and harvest. Significance levels () of the F-tests are shown in Table 7. A significance level less than 0.01 indicates that a factor has a statistically significant effect on the measured property at the 99.0% of probability.

An additional fertilisation had not any statistically significant effect on the volatile matter, calorific value, or N, S, C, H, and ash contents (Table 7), which is in agreement with the results obtained by other authors [4]. Results from chlorine have not been considered in the ANOVA from Table 7 due to an important proportion of the chlorine values are lower than 0.01%.

Likewise, no differences regarding volatile matter, H, N and S have been found between the biomass obtained from clones I-214 and AF2 (see Table 7). Clone I-214 exhibited a slightly higher ash content (mean value 2.9% db) than clone AF2 (mean value 2.5% db). The effect of the ash can generate changes in the carbon content and calorific values, which have statistical differences. In fact, by recalculating carbon content and calorific values to ash free data, it can be demonstrated that carbon and calorific values are equals for both clones I-214 and AF2 with significance values far higher than 0.01.

The very low significance levels (see Table 7) indicate that the rotation cycle or the root/stem age had a statistically significant effect on every studied property. In general, calorific values decrease with the number of rotation cycles (see Figure 4) probably due to the highest ash contents in R9S3. Nevertheless, by comparing the data in Figure 4 with Figure 5, it can be seen that the ash content is similar among R1S1A, R3S2A, R3S3B or R6S3B, even though the calorific value is higher in R1S1A. The highest bark to wood ratio of younger trees could explain the higher calorific values of 1-year old trees (R1S1A), as bark is known for having energy-rich compounds such as lignin, resins and oils [4,22]. However, the energy content of poplar does not justify the performance of harvests, which have maximum variations not exceeding 4%, e. g. between R9S3A and R1S1A, a very low value by comparing with biomass yield, that varied greatly between harvests (between some harvests more than 50%, see Figure 3). In summary and from energy point of view, the biomass yield obtained is the main variable for performing harvests.

Mean ash content (Figure 5) was 2.4 wt-% during the first two rotation cycles. The sharp increase (3.3 wt-%) observed during the third rotation cycle (R9S3) seems to be a consequence of a mineral concentration due to the proliferation of number of branches and twigs as a function of the stem/root age. The highest bark to wood ratio of the branches and twigs generates a higher proportion of ash in the R9S3 poplar. Volatile matter (Figure 5) does

not seem to follow any clear tendency.

The N content in 1-year old trees was significantly higher than the values obtained for older trees (see Figure 6). It is widely accepted that nutrient concentrations are lower in older and bigger deciduous trees than in smaller and younger ones [4, 24-25]. The higher N contents found in 1-year old trees demonstrate that harvesting very young plantations should be avoided, not only to minimise nutrient removal from the site, but also to avoid the use of a biomass with high contents of undesirable elements from a combustion quality point of view. The nitrogen and sulphur (Fig. 5) increased during the third rotation cycle (R9S3). One explanation of this fact is analogous to that given in the ash content behaviour as a function of the root/stem age, i. e., the shift in proportions of different fractions of the tree (twigs/branches/stem) with increasing age, and consequently the different bark to wood ratio, are likely to affect the quality of the biomass, as finally happens.

3.2.2 Ash analyses and ash melting behaviour

Ash composition during three rotation cycles is shown in Table 8 as a function of the fertilisation and the harvesting scheme used (A or B). Results are expressed as oxides on ash basis. Contents of titanium oxide (results not shown) are generally below the quantification limit (0.002% dry basis). In Table 9, the content of the elements recalculated on biomass is listed.

Significant levels for the three considered factors are shown in Table 10. It is important to note that the chemical composition of the ashes obtained from trees which received a supplementary fertilisation did not differ from the ones receiving only a basic dose.

Some differences were found between clones (Table 10). Calcium and phosphorous concentrations were significantly higher in I-214 clone. Potassium did not vary with the type of clone at the 99% probability (α =0.01), although K would show differences in the type of clone if the statistical contrast was carried out at the 95% probability (α =0.05). Tharankan et al. found significant differences in Ca, P, K, and Mg contents when studying the stems (with bark) of 7 different poplar clones, showing that natural clonal variation influences ash composition [7].

Most of the constituent elements of the ashes (Al, Ca, Fe, K, Na and Si) were affected by the rotation cycle and/or harvesting scheme used (Table 10). Figure 7 displays their temporal trends. Ca was found to be the major element of poplar ashes and its temporal trend seems to follow the ash pattern.

It has been speculated that silicon has a structural function, conferring resistance to the cell walls of the epidermis. Higher values of Si were found in trees with older roots (Figure 7). On the contrary, aluminium and iron (Figure 7) decreased as a function of the tree age. Trace elements were well below the limits specified in the ISO 17225-2 standard (data not shown) [9].

The estimation of the degree of slagging and fouling of biomass ashes is of crucial significance due to the ash sintering problems. The deformation temperature (DT) in oxidising atmosphere, which can be determined by optical heating microscopy (CEN/TS 15370-1) [26] is often used as a temperature of reference to indicate the ash melting starting point [27-29].

No important differences were observed among poplar samples (results not shown), as the deformation temperature for most of them exceeded 1,450 °C, which is the maximum temperature reached by the instrument.

To overcome this limitation, the relation between alkaline earth oxides and alkaline oxides (R) was used to predict the ash melting behaviour [29]. The higher the alkaline earth to alkaline oxides ratio, the higher the sintering temperature. In general, it could be said that biomasses with R values higher than 2 should not present risk of sintering. This ratio is calculated according to the following equation:

$$\mathbf{R} = (\mathbf{C}\mathbf{a}\mathbf{O} + \mathbf{M}\mathbf{g}\mathbf{O}) / (\mathbf{K}_2\mathbf{O} + \mathbf{N}\mathbf{a}_2\mathbf{O})$$

The results of the ratio between alkaline earth oxides and alkaline oxides are plotted in Figure 8. 1-year old trees presented the lowest ratios, as a consequence of its high K content and its low Ca content in comparison to 2 to 3-year old trees (see Figure 7). Ratios above 3.0 were obtained for trees older than 1 year, suggesting that this biomass should not cause important sintering problems when combusted. No statistically significant effect in R was obtained regarding fertilisation dose and clone type.

Examples of the good combustion behaviour of poplar can be found in the literature [23]. In previous combustion tests performed in a bubbling fluidised bed combustor, neither visual bed agglomeration nor sintering was observed on the heat exchangers [23]. The good melting behaviour of fuels with high Ca content as opposed to those which exhibit high K content is due to the dilution effect caused by the adsorption of alkaline salts inside the pores and surfaces of Ca inorganic compounds, such as lime and portlandite pores [30].

4 CONCLUSIONS

This investigation leads to the following conclusions:

Yields around 9 dry tons per hectare and year were obtained in this marginal land with a poor sandy soil during the first and second rotation cycles when plots received a supplementary fertilisation. A supplementary fertilisation led to higher yields. Biomass production dropped severely in the third rotation cycle. The yield in the third rotation cycle was around 4 tons of dried matter per hectare and year, therefore the productive life of the crop, under the conditions tested (adverse soil and climate, high density, etc.), could be finished.

Poplar biomass from SRC can be considered a suitable solid biofuel due to its appropriate ash melting behaviour with relations between alkaline earth oxides and alkaline oxides higher than 3, and its low content of nitrogen, sulphur and chlorine. Averaged contents in dry basis of 0.44 wt-%, 0.03 wt-% and around 0.01 wt-% in nitrogen, sulphur and chlorine were obtained in this study, respectively.

Taking into account the basic results for more than 15 elements analysed such as N, S, Cl, C, H, Ca, Mg, K, Na, P, Fe, Mn, Zn, Si and Al, no important significance effect can be found in poplar depending on the additional fertilisation.

In general and excluding the R1S1 harvest (1-year-old root and stem), the quality of the

biomass obtained from the first and second rotation cycles was similar, and it differed in some parameters from the quality of the biomass from the third rotation cycle. For example, averaged ash content in dry basis of 3.3 wt-% was obtained in the third rotation cycle in comparison with 2.3 wt-% and 2.5 wt-% obtained for the first and second cycles, respectively.

1-year old trees exhibited higher calorific values but also significantly higher N contents. Under the specific conditions tested in this study, an additional cut during the first rotation cycle when trees are 1-year old is not justified by either biomass quality or yield. In this context and applying additional fertilisation, the plots where biomass was not harvested the first year (harvest alternative B) provided higher accumulated yields, 69 dry Mg ha⁻¹, as opposed to 58 dry Mg ha⁻¹ (harvest alternative A). This difference was still higher when only basic fertilisation was applied.

Both clones (AF2 and I-214) seem to be similarly and well adapted to the harsh edaphoclimatic conditions of this region. Energy crops from both clones provided similar biomass quality and yields.

5 REFERENCES

[1] Communication from the Commission. Renewable Energy Road Map Renewable energies in the 21st century: building a more sustainable future. Brussels: European Commission COM; 2006.

[2] Communication from the Commission. Green paper: A 2030 framework for climate and energy policies. Brussels: European Commission COM; 2013.

[3] S.Y. Searle, C.J. Malins, Will energy crop yields meet expectations?, Biomass and Bioenergy 65 (2014) 3-12.

[4] D. Kauter, I. Lewandowski , W. Claupein, Quantity and quality of harvestable biomass from *Populus* short rotation coppice for solid fuel use—a review of the physiological basis and management influences, Biomass and Bioenergy 24 (2003) 411-427.

[5] S. Fang, X. Zhai, J. Wana, L. Tang, Clonal variation in growth, chemistry and calorific value of new poplar hybrids at nursery stage, Biomass and Bioenergy 54 (2013) 303-311.

[6] W.A. Kenney, A review of biomass quality research relevant to the use of poplar and willow for energy conversion, Biomass and Bioenergy 21 (1990) 163-188.

[7] P.J. Tharankan, T.A. Volk, L.P. Abrahamson, E.H. White, Energy feedstock

characteristics of willow and hybrid poplar clones at harvest age, Biomass and Bioenergy 25 (2003) 571-580.

[8] V.L. Goel, H.M. Behl, Fuelwood quality of promising tree species for alkaline soil sites in relation to tree age, Biomass and Bioenergy 10 (1996) 57-61.

[9] ISO 17225-2:2014, Solid biofuels—Fuel Specification and Classes- Part 2: Graded wood pellets. ISO International Organization for Standarization.

[10] ISO 17225-3:2014, Solid biofuels—Fuel Specification and Classes- Part 3: Graded wood briquettes. ISO International Organization for Standarization.

[11] ISO 17225-4:2014, Solid biofuels—Fuel Specification and Classes- Part 4: Graded wood chips. ISO International Organization for Standarization.

[12] P. Paris, L. Mareschi, M. Sabatti, A. Pisanelli, A. Ecosse, F. Nardin, G. Scarascia-Mugnozza, Comparing hybrid *Populus* clones for SRF across northern Italy after two biennial rotations: Survival, growth and yield, Biomass and Bioenergy 35 (2011) 1524-1532.

[13] H. Sixto, J. Salvia, M. Barrio, M.P. Ciria, I. Cañellas, Genetic variation and genotypeenvironment interactions in short rotation *Populus* plantations in Southern Europe, New Forests 42 (2) (2011) 163-177.

[14] P. Paris, L. Mareschi, M. Sabatti, T. Luca, G. Scarascia-Mugnozza, Nitrogen removal and its determinants in hybrid *Populus* clones for bioenergy plantations after two biennial rotations in two temperate sites in northern Italy, iForest-Biogeosciences and Forestry 8 (2015) 668-676.

[15] H. Sixto, P. Gil, P. Ciria, F. Camps, M. Sánchez, I. Cañellas, J. Voltas, Performance of hybrid poplar clones in short rotation coppice in Mediterranean environments: analysis of genotypic stability, Global Change Biology-Bioenergy 6 (2014) 661–671.

[16] P. Ciria, E. González, J.E. Carrasco. The effect of fertilization and planting density on biomass productivity of poplar harvested after three-years rotation. In: Proceedings of the 12th European Biomass Conference & Exhibition, Amsterdam, 2002, p. 283-286.

[17] P. Ciria, R. Barro, J. Pérez, E. Marcos, A. Moyano. Decomposition and dynamics of poplar leaves on short rotation conditions". In: Proceedings of the 17th European Biomass Conference & Exhibition, Hamburg, 2009, p. 502-506.

[18] P. Pérez, R. Barro, P. Ciria, L.S. Esteban, J.E. Carrasco. Behaviour of poplar stored outdoors as chips or stems, In: Proceedings of the 19th European Biomass Conference & Exhibition, Berlin, 2011, p. 770-777.

[19] ISO 17225-1:2014, Solid biofuels—Fuel Specification and Classes- Part 1: General requirements. ISO International Organization for Standarization.

[20] R.W. Bryers. Fireside slagging, fouling and high-temperature corrosion of heat transfer surface due to impurities in steam raising fuels, Progress in Energy and Combustion Science 22 (1996) 29-120.

[21] A.A. Khan, W. de Jong, P.J. Jansens, H. Spliethoff. Biomass combustion in fluidized bed boilers: Potential problems and remedies, Fuel Processing Technology 90 (2009) 21– 50.

[22] W.A. Kenney. A review of biomass quality research relevant to the use of poplar and willow ofr energy conversion, Biomass 21 (1990) 163-188.

[23] M.J. Fernández, R. Escalada, Influence of the amount of bed material on the distribution of biomass inorganic elements in a bubbling fluidised bed combustion pilot plant, Fuel 86 (2007) 867-876.

[24] L. Rytter, Nutrient content in stems of hybrid aspen as affected by tree age and tree size, and nutrient removal with harvest, Biomass and Bioenergy 23 (2002) 13-25.

[25] M-L Sander, T. Ericsson, Vertical distributions of plant nutrients and heavy metals in Salix viminalis stems and their implications for sampling, Biomass and Bioenergy 14 (1998) 57-66.

[26] CEN/TS 15370-1:2006, Solid Biofuels — Determination of ash melting behaviour – Part 1: Characteristic temperatures method. CEN European Committee for Standarization.

[27] C. Wilén, A. Moilanen, E. Kurkela. Biomass feedstock analyses 1996, VTT Publications 282, Espoo (Finland), 1996.

[28] B-J Skrifvars, M. Öhman, A. Nordin, M. Hupa, Predicting bed agglomeration tendencies for biomass fuels fired in FBC boilers: a comparison of three different methods, Energy and Fuels 13 (1999) 359-363.

[29] M.J. Fernández, J.E. Carrasco, Comparing methods for predicting the sintering of biomass ash in combustion, Fuel 84 (2005) 1893-1900.

[30] M.J. Fernández, P. Díaz, L. Gutiérrez, J.E. Carrasco, The effect of the addition of chemical materials on the sintering of biomass ash, Fuel 87 (2008) 2651-2658.

6 ACKNOWLEDGEMENTS

This work was part of a Spanish project to promote the use of poplar from short rotation coppice. It has been funded by the INIA project RTA2005-00182-C02-01 (Subprogramme of National Resources and Agricultural Technologies in coordination with the Autonomous Regions) supported by European Regional Development Funds (ERDF).

| Year | AMT (°C) | AmT (°C) | MMT (°C) | MmT (°C) | MT (°C) | P (1 m ⁻²) | Solar radiation (MWh m ⁻²) | Relative humidity (%) |
|------|-------------|----------|-------------|-------------|---------|------------------------|---|--------------------------|
| 2006 | 36.0 | -13.5 | 17.9 | 5.6 | 11.6 | 554 | 1.27 | 69 |
| 2007 | 33.8 | -7.4 | 16.8 | 5.1 | 10.0 | 342 | 1.43 | 68 |
| 2008 | 34.9 | -7.8 | 16.6 | 4.1 | 9.9 | 649 | 1.48 | 71 |
| 2009 | 34.7 | -14.0 | 17.7 | 4.4 | 10.8 | 390 | 1.60 | 65 |
| 2010 | 33.4 | -11.0 | 15.6 | 4.2 | 9.6 | 599 | 1.51 | 68 |
| 2011 | 35.7 | -11.9 | 17.9 | 4.9 | 11.1 | 380 | 1.63 | 66 |
| 2012 | 37.0 | -10.3 | 17.7 | 4.5 | 10.6 | 344 | 1.66 | 63 |
| 2013 | 33.8 | -8.8 | 16.5 | 3.2 | 9.5 | 595 | 1.55 | 70 |
| 2014 | 33.3 | -6.7 | 18.0 | 3.6 | 10.7 | 596 | 1.60 | 69 |

Table 1: Weather conditions measured at the experimental site.

AMT: Absolute maximum temperature, AmT: Absolute minimum temperature, MMT: Mean maximum temperature, MT: Mean temperature, P: Precipitation.

| Harvesting alternative | Clone | Fertilisation | Code | Age of tro | ees (years) |
|------------------------|-------|---------------|---------------|----------------------|-------------|
| | | | | Root | Stem |
| | I-214 | BF | | | |
| А | 1-214 | BF + AF | R1S1A | 1 | 1 |
| | AF2 | BF | | - | - |
| | 1112 | BF + AF | | | |
| | I-214 | BF | | | |
| А | | BF + AF | R3S2A | 3 | 2 |
| | AF2 | BF | 100211 | 5 | - |
| | | BF + AF | | | |
| | I-214 | BF | | | |
| А | | BF + AF | R6S3A | 6 | 3 |
| | AF2 | BF | 1000011 | 0 | 5 |
| | | BF + AF | | 583A 6 3 983A 9 3 | |
| | I-214 | BF | | | |
| А | | BF + AF | R9S3A | 9 | 3 |
| 21 | AF2 | BF | 105511 | , | 5 |
| | 1112 | BF + AF | | | |
| | I 214 | BF | | | |
| P | 1-214 | BF + AF | R383 B | 3 | 3 |
| D | 4 5 2 | BF | K555D | 5 | 5 |
| | ΑΓ2 | BF + AF | | | |
| | I 214 | BF | | | |
| P | 1-214 | BF + AF | D683B | 6 | 3 |
| D | 4.52 | BF | RUSSD | 0 | 5 |
| | ΑΓΔ | BF + AF | | | |
| | I 214 | BF | | | |
| р | 1-214 | BF + AF | DOS3B | 0 | 3 |
| D | 4 52 | BF | N933D | 9 | |
| | ΑΓΖ | BF + AF | | | |

Table 2: Variables considered (two harvesting alternatives, two clones and two fertilization rates) and codes used (different root/stem ages) in the experiments.

Alternative A: with cutting after the 1st year. Alternative B: without cutting after the 1st year. BF: basic fertilisation. AF: additional fertilisation

Table 3: Fertiliser applications. A basic fertilisation (BF) was applied to half of the plots. The other half received a supplementary fertilisation (BF+AF).

Control plot

| | Harv | vesting alternati | ive A | | Harvesting alternative B | | | | | | |
|------------------------|---------------|-------------------|----------|---------|--------------------------|----------|---------|--|--|--|--|
| Year | R1S1A | R3S2A | R6S3A | R9S3A | R3S3B | R6S3B | R9S3B | | | | |
| 2006 | BF Cutting | | | | BF | | | | | | |
| 2007 | | BF | | | | _ | | | | | |
| 2008 | | Cutting | - | | Cutting | - | | | | | |
| 2009 | | | BF | | | BF | | | | | |
| 2010 | | | | | | | | | | | |
| 2011 | | | Cutting | | | Cutting | | | | | |
| 2012 | | | | | | | | | | | |
| 2013 | | | | | _ | | | | | | |
| 2014 | | | | Cutting | _ | | Cutting | | | | |
| TOTAL N:P:K (FU/ha) | 48:40:75 | 27:30:75 | 48:40:75 | | 48:40:75 | 48:40:75 | | | | | |

Plot with additional fertilisation

| | Harv | vesting alternati | ive A | | Harve | esting alternative | e B |
|------------------------|------------------|-------------------|------------|----------|-------------|--------------------|----------|
| Year | R1S1A | R3S2A | R6S3A | R9S3A | R3S3B | R6S3B | R9S3B |
| 2006 | BF+AF Cutting | | | | BF+AF | | |
| 2007 | | BF+AF | _ | | AF | | |
| 2008 | | Cutting | - | | Cutting | | |
| 2009 | | | BF+AF | | | BF+AF | - |
| 2010 | | | AF | | | AF | - |
| 2011 | | | Cutting | | | Cutting | - |
| 2012 | | | | | | | - |
| 2013 | | | - | AF | _ | | AF |
| 2014 | | | - | Cutting | _ | | Cutting |
| TOTAL N:P:K (FU/ha) | 144:82:188 | 123:72:188 | 199:91:209 | 88:32:40 | 240:124:301 | 199:91:209 | 88:32:40 |

| | a vesting se | | Harvesting a | alternative A | | , per tree (in Ha | rvesting alternativ | в): е В |
|--------|---------------|-------------|--------------|---------------|---------------|----------------------|---------------------|--------------|
| Clone | Fertilisation | R1S1A | R3S2A | R6S3A | R9S3A | R3S3B | R6S3B | R9S3B |
| 1.04.4 | BF | 60±38 (36) | 280±130 (36) | 100±47 (36) | 170±130 (33) | 590±360 (35) | 300±180 (34) | 200±140 (32) |
| I-214 | BF+AF | 110±49 (34) | 640±240 (34) | 640±410 (34) | 250±220 (34) | 940±390 (35) | 890±390 (34) | 380±160 (34) |
| | BF | 65±40 (36) | 350±180 (36) | 250±210 (36) | 150±120 (33) | 910±670 (34) | 470±380 (33) | 540±420 (32) |
| AF2 | BF+AF | 130±80 (34) | 670±320 (34) | 920±770 (34) | 510± 410 (34) | 820±640 (33) | 1,000±840 (33) | 860±650 (32) |

Table 4: Averaged mass recorded per tree as a function of root/stem age following the two harvesting schemes (A and B). Mean \pm standard deviation in g per tree (number of trees).

| Fertilisation | Root age | Alternative A | Alternative B |
|----------------|----------|---------------|---------------|
| Basic | R3S3 | 13 | 24 |
| Basic | R6S3 | 5.9 | 12 |
| Basic | R9S3 | 2.8 | 8.8 |
| Accumulated to | otal | 21 | 44 |
| Additional | R3S3 | 24 | 28 |
| Additional | R6S3 | 24 | 28 |
| Additional | R9S3 | 10 | 13 |
| Accumulated to | otal | 58 | 69 |

Table 5: Partial yield (dry Mg ha⁻¹) for each rotation cycle as a function of harvest alternative and fertilisation.

Table 6: Averaged values of physical and chemical characterisation of the two poplar clones depending on fertilisation, and root/stem age following the two harvesting schemes (A and B).

| Age | Clone | Fertilisation | Moisture | Ash | VM | Carbon | Hydrogen | Nitrogen | Sulphur | Chlorine | GCV | NCV |
|-------|-------|---------------|----------|--------|--------|--------|----------|----------|---------|----------|----------------------|----------------------|
| | | | (% wb) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (MJ kg ⁻¹ | (MJ kg ⁻¹ |
| | | | | | | | | | | | db) | db) |
| R1S1A | I-214 | additional | 60.0 | 2.9 | 79.6 | 49.8 | 6.3 | 0.69 | 0.04 | < 0.01 | 19.73 | 18.44 |
| R1S1A | AF2 | additional | 59.7 | 2.5 | 80.5 | 49.8 | 6.3 | 0.65 | 0.04 | < 0.01 | 19.89 | 18.60 |
| R1S1A | I-214 | basic | 58.6 | 2.2 | 79.7 | 50.1 | 6.3 | 0.58 | 0.03 | < 0.01 | 19.86 | 18.57 |
| R1S1A | AF2 | basic | 58.3 | 2.5 | 80.1 | 50.1 | 6.2 | 0.62 | 0.03 | < 0.01 | 19.87 | 18.60 |
| R3S2A | I-214 | additional | 54.5 | 2.7 | 80.9 | 48.2 | 6.1 | 0.21 | 0.03 | 0.01 | 19.55 | 18.29 |
| R3S2A | AF2 | additional | 54.7 | 2.2 | 79.8 | 48.2 | 6.1 | 0.33 | 0.03 | 0.01 | 19.55 | 18.46 |
| R3S2A | I-214 | basic | 52.7 | 2.4 | 80.0 | 48.3 | 6.1 | 0.34 | 0.03 | 0.01 | 19.55 | 18.28 |
| R3S2A | AF2 | basic | 53.0 | 2.2 | 80.9 | 48.6 | 6.1 | 0.34 | 0.03 | 0.02 | 19.71 | 18.29 |
| R3S3B | I-214 | additional | 52.0 | 2.3 | 81.8 | 48.8 | 6.2 | 0.35 | 0.03 | 0.02 | 19.61 | 18.33 |
| R3S3B | AF2 | additional | 53.1 | 2.1 | 81.3 | 48.9 | 6.1 | 0.48 | 0.04 | 0.02 | 19.72 | 18.46 |
| R3S3B | I-214 | basic | 51.3 | 2.4 | 82.2 | 48.8 | 6.1 | 0.22 | 0.03 | 0.01 | 19.60 | 18.34 |
| R3S3B | AF2 | basic | 52.9 | 2.2 | 82.8 | 48.7 | 6.2 | 0.27 | 0.03 | 0.02 | 19.61 | 18.33 |
| R6S3B | I-214 | additional | 54.1 | 2.3 | 81.8 | 49.3 | 6.0 | 0.40 | 0.03 | < 0.01 | 19.58 | 18.27 |
| R6S3B | AF2 | additional | 54.5 | 2.4 | 81.9 | 49.2 | 6.0 | 0.42 | 0.03 | 0.01 | 19.59 | 18.27 |
| R6S3B | I-214 | basic | 53.9 | 2.5 | 80.9 | 49.1 | 6.0 | 0.43 | 0.03 | < 0.01 | 19.61 | 18.29 |
| R6S3B | AF2 | basic | 54.7 | 2.7 | 80.4 | 49.1 | 6.0 | 0.43 | 0.03 | < 0.01 | 19.56 | 18.26 |
| R9S3A | I-214 | additional | 51.2 | 3.6 | 80.1 | 48.0 | 6.0 | 0.46 | 0.04 | < 0.01 | 19.31 | 18.00 |
| R9S3A | AF2 | additional | 54.1 | 3.0 | 81.4 | 48.6 | 6.0 | 0.46 | 0.04 | < 0.01 | 19.38 | 18.06 |
| R9S3A | I-214 | basic | 49.2 | 4.1 | 79.9 | 47.9 | 6.0 | 0.49 | 0.04 | < 0.01 | 19.20 | 17.90 |
| R9S3A | AF2 | basic | 50.8 | 3.6 | 80.2 | 48.1 | 6.0 | 0.50 | 0.04 | < 0.01 | 19.24 | 17.93 |
| R9S3B | I-214 | additional | 50.7 | 3.5 | 80.4 | 47.9 | 6.0 | 0.49 | 0.04 | 0.0 | 19.15 | 17.84 |
| R9S3B | AF2 | additional | 53.9 | 2.3 | 81.5 | 48.5 | 6.0 | 0.43 | 0.04 | 0.0 | 19.44 | 18.12 |
| R9S3B | I-214 | basic | 46.5 | 3.4 | 80.4 | 47.9 | 6.0 | 0.47 | 0.04 | < 0.01 | 19.17 | 17.87 |
| R9S3B | AF2 | basic | 52.8 | 2.6 | 81.5 | 48.5 | 6.0 | 0.48 | 0.04 | < 0.01 | 19.42 | 18.12 |
| | | average | 53.6 | 2.7 | 80.8 | 48.8 | 6.1 | 0.44 | 0.03 | nd | 19.54 | 18.25 |
| | | rsd (%) | 5.9 | 20.9 | 1.1 | 1.4 | 1.5 | 27.9 | 14.8 | nd | 1.1 | 1.2 |

VM: volatile matter; wb: wet basis; db: dry basis; GCV: gross calorific value at constant volume; NCV: net calorific value at constant pressure. nd: not determined. Results from R6S3A harvest are not analysed

Table 7: Significance levels from F-tests for the studied factors (clone, fertilisation and root/stem age following two harvest alternatives (A and B)).

| | Ash | VM | Carbon | Hydrogen | Nitrogen | Sulphur | GCV | NCV | | |
|----------------------|---------|---------|---------|----------|----------|---------|---------|---------|--|--|
| Clone | 0.001 | 0.033 | 0.004 | 0.625 | 0.140 | 0.271 | < 0.001 | < 0.001 | | |
| Fertilisation | 0.370 | 0.361 | 0.897 | 0.140 | 0.260 | 0.581 | 0.941 | 0.134 | | |
| Age | < 0.001 | < 0.001 | < 0.001 | < 0.001 | < 0.001 | < 0.001 | < 0.001 | < 0.001 | | |
| VM: volatile matter. | | | | | | | | | | |

| Age | Clone | Fertilisation | Al_2O_3 | CaO | Fe ₂ O ₃ | K ₂ O | MgO | MnO ₃ | Na ₂ O | P_2O_5 | SiO ₂ | ZnO |
|-------|-------|---------------|-----------|--------|--------------------------------|------------------|--------|------------------|-------------------|----------|------------------|--------|
| | | | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) |
| R1S1A | I-214 | additional | 0.26 | 36 | 0.16 | 16 | 6.4 | 1.68 | 0.12 | 6.3 | 1.7 | 0.18 |
| R1S1A | AF2 | additional | 0.25 | 34 | 0.20 | 19 | 6.6 | 1.51 | 0.23 | 6.9 | 2.1 | 0.17 |
| R1S1A | I-214 | basic | 0.38 | 33 | 0.20 | 19 | 7.4 | 1.55 | 0.11 | 6.7 | 2.2 | 0.23 |
| R1S1A | AF2 | basic | 0.24 | 35 | 0.19 | 16 | 6.6 | 2.16 | 0.14 | 5.9 | 2.5 | 0.20 |
| R3S2A | I-214 | additional | 0.33 | 36 | 0.18 | 17 | 3.9 | 0.49 | 0.23 | 6.8 | 2.4 | 0.09 |
| R3S2A | AF2 | additional | 0.35 | 31 | 0.20 | 22 | 4.3 | 0.53 | 0.40 | 7.7 | 3.1 | 0.12 |
| R3S2A | I-214 | basic | 0.30 | 34 | 0.15 | 21 | 3.8 | 0.47 | 0.21 | 6.7 | 1.9 | 0.15 |
| R3S2A | AF2 | basic | 0.38 | 36 | 0.19 | 22 | 6.0 | 0.54 | 0.38 | 8.8 | 3.2 | 0.18 |
| R3S3B | I-214 | additional | 0.34 | 43 | 0.17 | 18 | 4.2 | 0.97 | 0.45 | 8.9 | 3.5 | 0.16 |
| R3S3B | AF2 | additional | 0.42 | 33 | 0.22 | 18 | 5.6 | 0.77 | 0.35 | 7.3 | 5.1 | 0.21 |
| R3S3B | I-214 | basic | 0.31 | 41 | 0.15 | 14 | 3.5 | 0.60 | 0.36 | 5.8 | 2.7 | 0.14 |
| R3S3B | AF2 | basic | 0.36 | 36 | 0.30 | 17 | 4.5 | 0.73 | 0.51 | 6.5 | 3.9 | 0.18 |
| R6S3B | I-214 | additional | 0.17 | 39 | 0.26 | 14 | 5.6 | 0.89 | 0.38 | 6.0 | 3.5 | 0.14 |
| R6S3B | AF2 | additional | 0.15 | 41 | 0.24 | 12 | 6.0 | 0.78 | 0.39 | 6.0 | 3.4 | 0.14 |
| R6S3B | I-214 | basic | 0.17 | 40 | 0.44 | 13 | 5.1 | 0.59 | 0.21 | 5.9 | 2.8 | 0.15 |
| R6S3B | AF2 | basic | 0.17 | 43 | 0.25 | 11 | 5.2 | 0.54 | 0.28 | 5.4 | 3.5 | 0.17 |
| R9S3A | I-214 | additional | 0.10 | 44 | 0.11 | 12 | 4.8 | 0.38 | 0.19 | 5.6 | 2.6 | 0.14 |
| R9S3A | AF2 | additional | 0.11 | 41 | 0.09 | 12 | 4.8 | 0.26 | 0.17 | 5.3 | 2.4 | 0.15 |
| R9S3A | I-214 | basic | 0.09 | 44 | 0.07 | 10 | 4.1 | 0.19 | 0.22 | 5.3 | 2.4 | 0.13 |
| R9S3A | AF2 | basic | 0.12 | 41 | 0.08 | 11 | 5.2 | 0.28 | 0.17 | 4.9 | 2.4 | 0.12 |
| R9S3B | I-214 | additional | 0.10 | 46 | 0.07 | 10 | 4.5 | 1.37 | 0.14 | 5.8 | 3.1 | 0.10 |
| R9S3B | AF2 | additional | 0.13 | 41 | 0.09 | 12 | 4.9 | 0.50 | 0.20 | 5.4 | 3.3 | 0.18 |
| R9S3B | I-214 | basic | 0.10 | 45 | 0.08 | 11 | 3.8 | 1.00 | 0.15 | 6.1 | 3.0 | 0.11 |
| R9S3B | AF2 | basic | 0.11 | 41 | 0.08 | 11 | 5.3 | 0.40 | 0.15 | 5.2 | 3.2 | 0.16 |
| | | average | 0.23 | 39 | 0.17 | 15 | 5.1 | 0.80 | 0.26 | 6.3 | 2.9 | 0.15 |
| | | rsd (%) | 48.7 | 11.1 | 50.4 | 25.4 | 20.0 | 63.6 | 45.3 | 16.6 | 25.3 | 22.3 |

Table 8: Ash analyses as oxides and on ash basis (averaged values) of two poplar clones during 3 rotation cycles, depending on fertilisation and root/stem age following different harvesting schemes (A or B).

db: dry basis; nd: not determined.

Table 9: Ash analyses on biomass basis (averaged values) of two poplar clones during 3 rotation cycles, depending on fertilisation and root/stem age following different harvesting schemes (A or B).

| Age | Clone | Fertilisation | Al | Ca | Fe | K | Mg | Mn | Na | Р | Si | Ti | Zn |
|-------|-------|---------------|--------|--------|-----------|-----------|----------|----------|--------|--------|--------|----------|--------|
| | | | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) | (% db) |
| R1S1A | I-214 | additional | 0.0040 | 0.74 | 0.0033 | 0.38 | 0.111 | 0.0256 | 0.0026 | 0.080 | 0.023 | 0.0001 | 0.0041 |
| R1S1A | AF2 | additional | 0.0033 | 0.60 | 0.0036 | 0.40 | 0.098 | 0.0200 | 0.0042 | 0.075 | 0.024 | 0.0001 | 0.0033 |
| R1S1A | I-214 | basic | 0.0043 | 0.51 | 0.0030 | 0.35 | 0.095 | 0.0178 | 0.0018 | 0.063 | 0.022 | 0.0001 | 0.0041 |
| R1S1A | AF2 | basic | 0.0031 | 0.60 | 0.0033 | 0.33 | 0.096 | 0.0280 | 0.0026 | 0.064 | 0.029 | 0.0001 | 0.0039 |
| R3S2A | I-214 | additional | 0.0047 | 0.69 | 0.0034 | 0.37 | 0.063 | 0.0070 | 0.0046 | 0.081 | 0.030 | 0.0002 | 0.0020 |
| R3S2A | AF2 | additional | 0.0041 | 0.48 | 0.0031 | 0.40 | 0.057 | 0.0061 | 0.0065 | 0.075 | 0.032 | 0.0002 | 0.0021 |
| R3S2A | I-214 | basic | 0.0039 | 0.58 | 0.0025 | 0.41 | 0.054 | 0.0060 | 0.0037 | 0.071 | 0.021 | 0.0001 | 0.0029 |
| R3S2A | AF2 | basic | 0.0044 | 0.56 | 0.0030 | 0.41 | 0.079 | 0.0063 | 0.0062 | 0.085 | 0.033 | 0.0002 | 0.0032 |
| R3S3B | I-214 | additional | 0.0040 | 0.69 | 0.0027 | 0.34 | 0.058 | 0.0117 | 0.0076 | 0.089 | 0.037 | 0.0003 | 0.0028 |
| R3S3B | AF2 | additional | 0.0046 | 0.49 | 0.0032 | 0.31 | 0.070 | 0.0084 | 0.0053 | 0.066 | 0.050 | 0.0004 | 0.0035 |
| R3S3B | I-214 | basic | 0.0039 | 0.70 | 0.0026 | 0.27 | 0.051 | 0.0077 | 0.0065 | 0.061 | 0.031 | 0.0002 | 0.0027 |
| R3S3B | AF2 | basic | 0.0042 | 0.55 | 0.0045 | 0.30 | 0.059 | 0.0083 | 0.0081 | 0.062 | 0.040 | 0.0003 | 0.0031 |
| R6S3B | I-214 | additional | 0.0021 | 0.63 | 0.0042 | 0.26 | 0.076 | 0.0107 | 0.0063 | 0.060 | 0.037 | < 0.0002 | 0.0026 |
| R6S3B | AF2 | additional | 0.0019 | 0.68 | 0.0040 | 0.24 | 0.086 | 0.0098 | 0.0068 | 0.062 | 0.038 | < 0.0002 | 0.0027 |
| R6S3B | I-214 | basic | 0.0023 | 0.72 | 0.0077 | 0.28 | 0.076 | 0.0079 | 0.0039 | 0.065 | 0.033 | < 0.0002 | 0.0031 |
| R6S3B | AF2 | basic | 0.0025 | 0.82 | 0.0047 | 0.25 | 0.085 | 0.0078 | 0.0056 | 0.064 | 0.044 | < 0.0002 | 0.0037 |
| R9S3A | I-214 | additional | 0.0019 | 1.11 | 0.0027 | 0.35 | 0.103 | 0.0073 | 0.0050 | 0.088 | 0.043 | < 0.0002 | 0.0040 |
| R9S3A | AF2 | additional | 0.0017 | 0.87 | 0.0018 | 0.31 | 0.087 | 0.0042 | 0.0037 | 0.070 | 0.033 | < 0.0002 | 0.0037 |
| R9S3A | I-214 | basic | 0.0019 | 1.27 | 0.0020 | 0.35 | 0.099 | 0.0040 | 0.0065 | 0.095 | 0.046 | < 0.0002 | 0.0043 |
| R9S3A | AF2 | basic | 0.0023 | 1.04 | 0.0020 | 0.33 | 0.111 | 0.0054 | 0.0044 | 0.077 | 0.040 | < 0.0002 | 0.0035 |
| R9S3B | I-214 | additional | 0.0019 | 1.16 | 0.0018 | 0.31 | 0.095 | 0.0257 | 0.0037 | 0.090 | 0.052 | < 0.0002 | 0.0027 |
| R9S3B | AF2 | additional | 0.0015 | 0.67 | 0.0014 | 0.23 | 0.066 | 0.0060 | 0.0033 | 0.054 | 0.035 | < 0.0002 | 0.0033 |
| R9S3B | I-214 | basic | 0.0019 | 1.08 | 0.0018 | 0.31 | 0.078 | 0.0180 | 0.0038 | 0.091 | 0.048 | < 0.0002 | 0.0029 |
| R9S3B | AF2 | basic | 0.0016 | 0.77 | 0.0015 | 0.24 | 0.083 | 0.0055 | 0.0030 | 0.061 | 0.039 | < 0.0002 | 0.0033 |
| | | average | 0.0030 | 0.75 | 0.0031 | 0.32 | 0.081 | 0.0110 | 0.0048 | 0.073 | 0.036 | nd | 0.0032 |
| | | rsd (%) | 37.8 | 30.1 | 43.8 | 17.2 | 22.6 | 66.1 | 35.3 | 16.5 | 23.9 | nd | 19.0 |
| | | | | C | ib: dry b | asis; nd: | not dete | ermined. | | | | | |

Table 10: Significance levels from ANOVA of ash analyses for the three studied factors (type of clone, fertilisation and root/stem age following two harvest alternatives (A and B)).

| | Al | Ca | Fe | K | Mg | Mn | Na | Р | Si | Zn |
|---------------|---------|---------|---------|---------|-------|-------|-------|---------|---------|-------|
| Clone | 0.527 | < 0.001 | 0.153 | 0.016 | 0.645 | 0.025 | 0.536 | < 0.001 | 0.474 | 0.724 |
| Fertilisation | 0.446 | 0.099 | 0.416 | 0.876 | 0.577 | 0.493 | 1.000 | 0.525 | 0.773 | 0.070 |
| Age | < 0.001 | < 0.001 | < 0.001 | < 0.001 | 0.030 | 0.024 | 0.004 | 0.070 | < 0.001 | 0.034 |



Figure 1: Experimental plot for each harvest alternative. BF: basic fertilisation. AF: additional fertilisation



Figure 2: Harvesting schemes. RS codes denote the age of root (R) and stems (S), in years, at the moment of the harvest



Figure 3: Dried biomass yield (means and LSD intervals) obtained depending on fertilisation and root/stem age, following two harvest alternatives (A or B): basic fertilisation (left) and additional fertilisation (right).



Figure 4: Gross and net calorific value (means and LSD intervals) during 3 rotation cycles and depending on root/stem age following two harvest alternatives (A and B).



Figure 5: Ash content and volatile matter (means and LSD intervals) during 3 rotation cycles and depending on root/stem age following two harvest alternatives (A and B).



Figure 6: Nitrogen and sulphur contents (means and LSD intervals) during 3 rotation cycles and depending on root/stem age following two harvest alternatives (A and B).



Figure 7: Elemental content evolution (means and LSD intervals) in trees with different root/stem age obtained following two harvest schemes (A or B) during 3 rotation cycles.



Figure 8: Relation between alkaline earth oxides and alkaline oxides (means and LSD intervals) depending on the root/stem age following two harvest schemes (A or B) during 3 rotation cycles.