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2 1 **Production and composition of biomass from short rotation**

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5 2 **coppice in marginal land: a 9-year study**

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28 11 **ABSTRACT:**

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32 13 The production and chemical composition of the biomass of four fast growing woody crops
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34 14 are studied across species and rotation cycles. High-density plantations of poplar, willow,
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36 15 black locust and sycamore were monitored for 9 years over 3-year rotation cycles in marginal
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38 16 land in the Mediterranean area. The fuel quality properties of the produced biomass was
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40 17 assessed using proximate and ultimate analysis, biomass and ash chemical composition (23
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42 18 elements), and ash melting behaviour.

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44 19 Willow, poplar and black locust energy crops produced 12, 9 and 7 dry Mg ha⁻¹ yr⁻¹ on
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46 20 average, indicating that these species can be well adapted to the unfavourable conditions of
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48 21 poor soils in low productivity areas in the Mediterranean region. In contrast, sycamore was
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50 22 not well adapted to the particular climate and soil conditions and provided very poor yields.
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52 23 The chemical composition of the biomass varied across species and rotation cycles. The
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54 24 results obtained from the chemical analysis of these woody species showed that they provided
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56 25 good quality for use in combustion processes, with low amounts of trace elements and

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2 26 chlorine (≈ 0.01 wt%), sulphur (≈ 0.04 wt%) and nitrogen (≈ 0.5 wt%), with the exception of
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4 27 black locust (N= 0.86 wt%). No sintering problems are expected during the combustion of the
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6 28 studied biomass. This study provides valuable information for the large-scale implementation
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9 29 of fast growing woody species dedicated to energy crops in low productivity areas of the
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11 30 Mediterranean region.

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16 32 Keywords: biomass, yield, woody species, quality, chemical analysis, combustion.
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26 36 **1 INTRODUCTION**

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30 39 The new EU Renewable Energy Directive (RED II) 2018/2001/EC [1] stipulates that 32% of
31 40 member states' total energy consumption should come from renewable sources by 2030,
32 41 increasing by 12% the target previously set in RED I (2009/28/EC) for 2020 [2]. In that
33 42 global scenario, where a balance between food provision, energy production and
34 43 environmental sustainability is needed, the use of marginal lands to grow energy crops has
35 44 very recently become a topic of interest for research [3]. Even though the concept of
36 45 "marginal land" can vary depending on the context [4], this term can be used to refer to low
37 46 quality agricultural areas that are unsuitable for producing food due to low productivity
38 47 caused by poor soils and/or unfavourable climates [5-6]. A previous study estimated that a
39 48 total area of 63.7 Mha in Europe is marginal land suitable for the production of bioenergy
40 49 resources, which represents less than 30% of overall marginal land in Europe [7]. In Spain, a
41 50 previous study identified about 2 Mha of arable marginal lands where traditional food crops
42 51 are not economically sustainable and could have bioenergy potential [8]. Most of them are
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2 51 low productivity areas whose winter cereal grain production is below 1.5 Mg ha⁻¹ due to poor
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4 52 soils with an organic matter content below 2%.

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9 54 Poplar and willow, among others, have been previously identified as potential energy crops
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11 55 for cultivation on marginal lands, as they are fast-growing species that can be adapted to the
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14 56 most unfavourable site conditions [3,9]. Poplar trees are quite abundant in the Iberian
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16 57 Peninsula, particularly in riverside plantations in temperate-cold or temperate climates from
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19 58 sea level up to 1500 m. Poplar is probably one of the most well-known and extensively used
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21 59 fast growing forest trees in short rotation intensive culture plantations dedicated to biomass
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24 60 production on an international basis. Species, clones and interspecific hybrids have been
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26 61 tested in multisite experiments all over the world [9]. Willows have a high potential for
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29 62 biomass production all over Europe. The willow is known to present favourable biological
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31 63 attributes in addition to having a higher tolerance than the poplar to a wide variety of climate
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34 64 and soil conditions, which is particularly important under the ensuing climatic change [10-
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In contrast to poplar and willow, the experience in Europe with sycamore and black locust for dedicated energy crops is very limited. Their great adaptability to water limitations make them highly attractive options for biomass production under Mediterranean conditions, particularly considering the predicted rise in summer temperatures over the 21st century, as foreseen by the Intergovernmental Panel on Climate Change (IPCC) in all of the assessed emission scenarios [12]. Plane trees are being extensively used in the Mediterranean region for ornamental purposes in urban areas and are characterised by their high growth rate and high sprouting capacity [13]. Consequently, the potential of sycamore as a bioenergy feedstock has attracted interest in Spain and a prediction model has even been developed to

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2 76 estimate pruned biomass obtained from sycamore in urban parks and green areas [14].
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4 77 However, using plane trees as a dedicated energy crop might have limited possibilities in the
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6 78 Mediterranean region, as preliminary studies suggest that sycamore in short rotation coppice
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9 79 (SRC) provide lower yields than other fast-growing forest trees under the same conditions
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11 80 [10,15]. On the other hand, black locust is another species that could also potentially be
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14 81 highly productive under Mediterranean climate conditions [16-18]. It is a nitrogen-fixing
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16 82 species that belongs to the Leguminosae family with a considerable resprouting capacity after
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19 83 cutting [19].
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24 85 Biomass production aside, literature regarding the composition and quality of the produced
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26 86 biomass is scarce, particularly in the case of emergent energy crops such as sycamore and
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29 87 black locust. In this scenario, there was an urgent need to further investigate the potential of
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31 88 these crops by taking into consideration not only their potential yields in the long term but
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33 89 also their properties. Even though woody biomass exhibits lower amounts of ash, N, Cl and
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36 90 other troublesome elements than herbaceous biomass, it might not be exempted from causing
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39 91 corrosion and sintering problems in combustion processes or negative environmental impacts
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41 92 [20-21]. Lately, the quality requirements of woody biofuels regarding ash, N, Cl, S, trace
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43 93 elements and other properties have been established in the international standard ISO 17225
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45 94 “Fuel Specifications and classes” for specific trade forms of solid biofuels such as pellets
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48 95 [22], briquettes [23], and wood chips [24]. Biomass from short rotation coppice, fast growing
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50 96 species characterised by high bark-to-wood ratios, could exceed the maximum values allowed
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53 97 in these quality requirements and that could limit their use in the residential sector and small-
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55 98 scale
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58 99 applications.
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2 100 The objective of this work is to evaluate biomass production and the combustion properties of
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4 101 the biomass of four fast growing woody species (poplar, willow, black locust and sycamore)
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6 102 produced under short rotation conditions across three 3-year rotation cycles in marginal land
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9 103 under Mediterranean conditions. The results obtained can be of great interest for the
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11 104 commercial implementation of dedicated energy crops in the Mediterranean region,
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14 105 particularly for low productivity agricultural areas.
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23 109 **2 MATERIALS AND METHODS**

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29 112 The following species and clones were used in this study: *Populus x euramericana* ('I-214'),
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32 114 *Salix matsudana x Salix* spp. ('Levante'), *Platanus hybrida* ('Girona'), and *Robinia*
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35 116 *pseudoacacia* ('Nyirsegi'). The poplar and willow clones studied have previously shown high
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61 146 62 147 **2.2 Climate**

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64 149 The plantation site was located at the Centre for the Development of Renewable Energy
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66 151 Sources (CEDER-CIEMAT) with coordinates 41° 36' North, 2° 30' West (Figure 1). This
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per year). Summers have mean temperatures around 18 °C and maximum temperatures well above 30 °C (see Table 1). Even though historical annual rainfall averages are about 500 mm [30], it is important to note that rainfall in this region can vary largely from year to year. As can be seen in Table 1, rainfall was below 400 mm in 2009, which is a value more typical of arid regions, whereas 600 mm of rainfall was recorded in 2010, 2013 and 2014. It is important to note that irrigation was applied based on crop requirements and weather conditions, using studies of potential evaporation, water use efficiency and water stress. The procedures used were established in other research projects [31]. Plots were drip irrigated during the dry months (from May to September), i.e. poplar and willow with 395.0 mm and black locust and sycamore with 193.5 mm on average.

Table 1: Weather conditions measured (annual and global mean values) and at the experimental site.

2.4 Soil

Table 2 shows the soil characteristics at the experimental site before planting. The soil has a sandy texture with more than 80% as sand, 35% of gravels, low organic matter ($\leq 0.6\%$), and also low contents of available P and K. It is a light and well-drained soil, with a very low retention capacity for water and nutrients, which could result in losses by leaching. For all these reasons, it can be considered a marginal, poor, low productive soil, which demands a fractionation of fertiliser applications to give time to the plant to absorb those nutrients and avoid losses by leaching.

Table 2: Soil characteristics at the experimental site.

2.5 Experimental design and agronomical practices

High-density plantations (10,000 trees per hectare, spaced 2.5 m × 0.4 m apart) of poplar, willow, black locust and sycamore were established with three replications in April 2009 (Figure 1).

Figure 1: Location and distribution of species and trees at the experimental plantation. Each black dot represents an individual tree.

Figure 1 also shows the distribution of species at the experimental location (left) and the distribution of trees per replicate subplot (right). Each replicate subplot consisted of 49 trees (black dots), resulting in square experimental units in which only the 9 central trees were monitored to avoid the border effect (Figure 1). Blank blocks in Figure 1 indicate plots belonging to other experimental trials. Manual planting was performed in all species. Cuttings were used for poplar and willow, and seedlings for black locust and sycamore.

Before planting, NPK fertilisation (8-15-15) was applied in April at 600 kg/ha at the experimental site. All plots, except for black locust, received additional fertilisation two months later with NPK (12-12-17) and microelements (0.1 wt% Fe; 0.02 wt% B and 0.01 wt% Zn) at 125 kg/ha, and with NPK (22-8-10) at 250 kg/ha in April 2011, 2013, and 2015. Deltamethryn 2.5 w/v-% was applied at the beginning of each vegetative period for beetle (*Melasoma populi*) control in poplar and willow plots.

2.6 Harvesting, sampling and analysis

To estimate biomass yields, 27 trees per species (9 trees per replication) were cut at the end of 2011, 2014 and 2017 (three different 3-year rotation cycles). The nine central trees of each

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2 175 replicate subplot were severed 10 cm above the ground at the end of each growth period
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4 176 (between December and January) and all of the aboveground biomass was used to determine
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6 177 biomass production. This biomass was also later used for analysis after sample preparation
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9 178 according to ISO 14780.

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11 179 Once biomass weight was determined, the collected biomass (9 trees per replicate subplot)
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14 180 was completely chipped and milled, and representative samples were obtained after
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16 181 homogenisation, dividing, drying, and grinding, as indicated in the ISO 14780 standard.
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18 182 Physical and chemical tests were performed in the CEDER-CIEMAT Laboratory of Biomass
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21 183 Characterisation according to the international standards for solid biofuels, which are listed in
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23 184 Table 3. Dry matter yields were determined by taking into account the weight of all the
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26 185 aboveground biomass before and after being oven-dried at 105 °C until constant weight.
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31 187 **Table 3:** Standards and analytical methods of the properties measured in the woody SRC.
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34 35 189 **2.7 Ash melting behaviour**

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38 190 Ash fusibility was based on the changes in shape detected during the heating of a cylindrical
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40 191 ash pellet (ash produced at 550 °C) from room temperature to 1450 °C in an air atmosphere.

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45 193 temperatures, following CEN/TS 15370-1 [32].
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50 195 The earth-alkaline to alkaline oxides ratio was used to predict the sintering behaviour of the
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52 196 studied fuels [33]. The performance of this index was evaluated elsewhere for different fuels,
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54 197 and it is calculated by using the following equation:
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$$57 \text{ Rake/ak} = (\text{CaO} + \text{MgO}) / (\text{K}_2\text{O} + \text{Na}_2\text{O})$$

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2 199 In general, biomass with Rake/ak indices higher than 2 should not present any risk of
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4 200 sintering at combustion temperatures of about 800 °C [33-34]. Below this value, the risk of
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6 201 sintering increases.
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10 11 203 **2.8 Statistical analysis**

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14 204 The statistical differences across species and rotation cycles on the production and the quality
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16 205 parameters of the produced biomass were evaluated by analyses of variance (ANOVA) using
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18 206 the Statgraphics Plus 5.0 software. Significant effects were revealed at $\alpha = 0.05$. Fisher's least
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21 207 significant difference (LSD) at the 95.0% confidence level was used to evaluate the
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32 33 212 **3 RESULTS AND DISCUSSION**

34 35 213 **3.1 Biomass yields**

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40 215 Biomass production is studied across species and rotation cycles (Figures 2). Willow provided
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42 216 the best yields (12 dry Mg ha⁻¹ yr⁻¹ as average), followed by poplar and black locust (8.9 and
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44 217 6.8 dry Mg ha⁻¹ yr⁻¹, respectively). Therefore, it seems that these woody species can be
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46 218 adapted relatively well to the harsh and unfavourable edaphoclimatic characteristics of
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48 219 marginal lands in the Mediterranean area.
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57 222 In contrast and even though the mortality of sycamore plants was zero, the biomass
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59 223 production of this species was extremely low, not even reaching 3 dry Mg ha⁻¹ yr⁻¹. Sycamore
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65 was not well adapted to the particular climate and soil conditions of the study site, and

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2 224 exhibited vegetative growing problems, probably in connection with the high altitude of the
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4 225 location (1099 m above sea level) and the significant temperature differences between day
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6 226 and night that characterised that area.
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11 228 Biomass production decreased significantly in the third rotation cycle for all species (Figure
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14 229 3), from 8-9 dry Mg ha⁻¹ yr⁻¹ on average during the first and second rotation cycles to 5 dry
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16 230 Mg ha⁻¹ yr⁻¹ in the third rotation cycle (Figure 2, right). The drop in biomass production in the
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19 231 third rotation cycle could be an indication of the end of the productive cycle of these species
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22 232 in arable marginal land, with willow production dropping over 50% between the first and the
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24 233 third rotations. By considering only the first and second rotation cycles, yields averaged 15.0,
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27 234 10.1, and 7.7 dry Mg ha⁻¹ yr⁻¹ for willow, poplar and black locust, respectively [9].
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33 **Figure 2:** Dried biomass yield (mean and LSD intervals) across species (left) and rotation
34 237 cycles (right).
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41 **Figure 3:** Dried biomass yield (Mg ha⁻¹ yr⁻¹) across species and rotation cycles.
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45 Other studies under marginal conditions reported similar biomass yields for black locust in
46 242 Germany (5.8 Mg ha⁻¹ yr⁻¹) [35], willow in North America, Northern Europe and Australia (5-
47 243 11 Mg ha⁻¹ yr⁻¹) [3], and poplar in Germany (7.6 Mg ha⁻¹ yr⁻¹) [36] and USA (6-15.8 Mg ha⁻¹
48 244 yr⁻¹) [3]. Stolarski et al. [37] obtained lower yields for black locust (1.6-2.0 Mg ha⁻¹ yr⁻¹) and
49 245 willow (5.1-9.1 Mg ha⁻¹ yr⁻¹) on poor soil in Poland after a 4-year rotation when no mineral
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51 246 fertilisers were applied, and similar yields were obtained for poplar (5.5-9.2 Mg ha⁻¹ yr⁻¹).
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53 247 Moreover, it is worth mentioning that productivity improved significantly when using soil
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55 248 amendments [16,35,37], which could be a viable option to increase the energy and economic
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2 249 value of short-rotation woody crops in low productive areas.
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6 251 Aravanopoulos reviewed the existing literature regarding the breeding of fast-growing forest
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11 253 and soil conditions [10], finding higher production rates in poplar and willow than in
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14 254 sycamore. This indicates that sycamore might not perform as well as other fast-growing
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16 255 species in some Mediterranean areas, particularly on marginal land with a continental
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18 256 influence, such as the area tested in this study.
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21 257 22 23 258 **3.2 Biomass characteristics and quality**

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26 259 In this section, the physical and chemical characteristics of the biomass obtained from the
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28 260 different fast-growing tree species across 3 rotation cycles are presented, and their fuel
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30 261 characteristics are evaluated. In addition, the combustion quality of the pellets and wood chips
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33 262 that could be produced from this biomass are discussed and compared to the requirements of
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36 263 ISO 17225-2 [22] and ISO 17225-4 [24] that grade wood pellets and chips, respectively.
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39 40 265 **3.2.1 Physical and chemical characteristics**

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43 266 Table 4 shows the results of the physical and chemical characterisation of poplar, black
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45 267 locust, willow and sycamore across rotation cycles. Moisture is generally high in SRC at
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47 268 harvest time. Mean moisture content for the trees studied in this work was around 40%, which
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49 269 indicates the need to introduce a drying step during the storage of the biomass, prior to its
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52 270 combustion or other thermochemical use.
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59 273 Ash content is an important property from a quality perspective, as it causes emissions in the
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2 274 to the ash layer that adheres to the tubes of the heat exchangers. Regardless of the species
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4 275 considered, the produced biomass exhibited 2-3 % ash. According to ISO 17225-2 [22], if this
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6 276 biomass was used to produce wood pellets, they would be classified as class I3 ($\leq 3\%$ ash) for
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9 277 industrial use. The wood chips produced from this raw material could be classified as class B1
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11 278 for commercial and residential applications (ISO 17225-4 [24]). The ash content of poplar,
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14 279 willow, black locust and sycamore is three to four times higher than pine sawdust, which is
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16 280 usually considered a high quality biofuel for the domestic sector.
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21 282 Corrosion in heat-exchanger tubes is not expected to be an issue, as all the species considered
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23 283 showed a low chlorine content ($<0.01-0.02\%$) and fulfilled all the requirements found in the
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26 284 applicable quality standards.
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31 286 S and N content in the fuel has been previously correlated with the formation of SO_2 and NO_x
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33 287 emissions during combustion [39]. As the emission of SO_2 and NO_x are the biggest cause of
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36 288 acid rain today, lower S and N contents in the fuel are desirable. Samples averaged 0.6 % N,
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38 289 within the range typically found for SRC biofuels [38]. The average S content in the produced
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41 290 biomass was relatively low ($\leq 0.05\text{ wt}\%$), and much lower than that the sulphur present in
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43 291 fossil fuels such as gas-oil. Directive 1999/32/EC establishes a S limitation of 0.1 % for gas-
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45 292 oil for domestic applications.
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51 **Table 4:** Proximate and ultimate analysis of the biomass across species and rotation cycles,
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54 and significance levels from ANOVA.
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59 298 The significance levels for the two factors considered are also shown in Table 4. The
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61 chemical composition of the biomass varied significantly across species and rotation cycles
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2 299 (Table 4, Figure 4). Black locust and poplar exhibited significantly higher calorific values
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4 300 (18.3-18.4 MJ/kg) than sycamore and willow (18.1-18.2 MJ/kg). Ash content also varied
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6 301 significantly across species, but mean values were maintained between 2.1% (black locust)
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9 302 and 2.6% (poplar).

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14 304 Although significant from a statistical point of view, the differences detected among species
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16 305 and rotations for GCV, NCV, C, and H are very small and with no practical relevance (Table
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18 306 4). As expected, GCV and C (figures not shown) followed the same trend as NCV. Net
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21 307 caloric values increased slightly from 18.2 MJ/kg in the first rotation cycles to 18.3 MJ/kg in
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23 308 the third rotation cycle. At the same time, ash also increased in the third rotation cycle (from
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25 309 2.3 % up to 2.6%). Bark to wood ratio is expected to increase in SRC as crops age due to the
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28 310 proliferation of small branches and twigs. Bark typically has higher ash content than wood,
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30 311 and it is also known to possess energy-rich compounds such as lignin, resins, and oils, which
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33 312 cause calorific value to increase [28].
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39 **Figure 4:** Net calorific value and ash content (mean and LSD intervals) across species (left)
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and rotation cycles (right).

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It is important to highlight the elevated N content of black locust (Figure 5) in comparison with the other species considered, as black locust is a legume that fixes N from the atmosphere through its root nodules. Black locust averaged 0.86% N while mean contents for poplar, sycamore and willow were between 0.43-0.53% N. The N content of the biomass was not dependent on the rotation cycle. S content was low, and it varied between 0.03 and 0.05%. In general, it seems that poplar and willow presented lower S content (0.03%) than black locust and sycamore (0.05%). No clear trend was observed for this element with the rotation

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6 326 **Figure 5:** Nitrogen and sulphur contents (mean and LSD intervals) depending on species
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9 327 (left) and rotation cycle (right).

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14 329 Taking into account the normative parameters and chemical composition of the biomass of the
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16 330 studied energy crops and by comparison with the current limits set in ISO 17225-2 [22] and -4
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18 331 [24] that grade wood pellets and chips, respectively, it could be said that the biomass from
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20 332 poplar, willow and sycamore plantations could be used as raw material to produce class B1
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22 333 wood chips for domestic and residential applications or class I3 wood pellets for industrial
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24 334 applications (ash content $\leq 3.0\%$). However, the biomass from black locust energy crops is
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26 335 not within the specifications of the international standard that grades wood pellets for
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28 336 industrial applications, since it exceeds the N requirements ($\leq 0.6\%$). This biomass could be
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30 337 used, in principle, to produce class B1 wood chips (ash content $\leq 3\%$).
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37
38 339 The interactions between species and rotations were not significant in the following
39
40 340 parameters: GCV, NCV, H, N and S (see Table 4). In contrast, ash did present interaction
41
42 341 because the ash content in all species was equal for the first and second rotation cycles. The
43
44 342 ash content was different among species in the third rotation cycle. It should be noted that ash
45
46 343 content as well as carbon did generate interaction at the 95% confidence level but not at 99%.
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48
49 344

50 345 **3.2.2 Ash analysis, ash recycling and ash melting behaviour**

51
52 346 The biomass ash of the species considered showed a similar chemical composition (Table 5),
53
54 347 where calcium and potassium are the major ash-forming elements, with global averages of
55
56 348 0.66 wt% and 0.28 wt%, respectively, followed by phosphorous (0.072 wt%), magnesium
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1
2 349 (0.064 wt%) and silicon (0.025 wt%). The rest of the elements were found at trace levels
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4 350 (<0.01 wt%).
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6 351
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9 352 **Table 5:** Chemical composition of the biomass across species and rotation cycles, and
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11 353 significance levels from ANOVA.
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16 355 Similar to Table 4, the significance levels for the two factors considered are shown in Table 5.
17
18 356 Ash chemical composition depended on the rotation cycle, although no clear pattern could be
19
20
21 357 derived. However, and similar to what was observed in the case of the ash content, Ca (Figure
22
23 358 6), the main ash-forming element, was higher in the biomass coming from the third rotation
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25
26 359 cycle in comparison with the previous ones. As mentioned above, the elevated ash content
27
28 360 levels detected in the biomass from the third rotation cycle were related to a higher bark to
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30
31 361 wood ratios. Calcium tends to be accumulated in the bark tissues in the form of oxalates and
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33 362 carbonates. K, Mg, and Si, which are also relevant elements from a quality perspective, did
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35
36 363 not vary with the rotation cycle (Table 5).
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41 365 Regarding the ash chemical composition across species (Figure 6-left and 7), it could be said
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43 366 that mean levels of ash, Ca, K, Si, and Mg tend to be generally higher for poplar and lower for
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45 367 black locust, although not always attaining significance. Other authors also found significant
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47
48 368 differences between the chemical composition of poplar and willow groups [40-41].
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53 370
54 **Figure 6:** Calcium content (mean and LSD intervals) depending on species (left) and rotation
55 371 cycle (right).
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Figure 7: K, Mg, Si, Mn, P, and Zn content (mean and LSD intervals) depending on species.

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2 374
3
4 375 In contrast to the proximate and ultimate analyses, interactions in chemical composition
5
6 376 between species and rotations were significant in most of the parameters: Ca, K, Si, Mg, P,
7
8 377 Al, Mn, Ti and Zn (see Table 4). In general, and similarly to ash content, calcium, the main
9
10 378 inorganic element in ash, generated interactions because its content was equal for the first and
11
12 379 second rotation cycles in all species, but different among species in the third rotation cycle.
13
14 380 Nevertheless, calcium did generate interaction at the 95% confidence level but not at 99%.
15
16 381

17
18 382 Trace elements were very low (Table 6) and always within the limits established for wood
19
20 383 pellets and wood chips, in ISO 17225-2 [22] and ISO 17225-4 [24] standards, respectively.
21
22 384 Biomass grown in uncontaminated soils is not expected to surpass the above-mentioned
23
24 385 requirements. The experimental area is located in the province of Soria, a region characterised
25
26 386 by a very low population density (<9 inhabitants per km²) due to the rural exodus in favour of
27
28 387 the cities and industrialised areas. Therefore, this rural, unpopulated, and under-industrialised
29
30 388 region, is not expected to be polluted with heavy metals as the ones that are being tested in
31
32 389 this section.
33
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35
36 391 **Table 6:** Trace elements of the biomass obtained from the short rotation woody crops during
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38 392 the third rotation cycle, and ISO 17225 requirements, parts 2 (woody pellets) and 4 (woody
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40 393 chips).
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43
44 395 In order to study the possibility of recycling the ashes generated in the combustion of the
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46 396 studied SRC and reusing them as crop fertilisers, the maximum content of trace elements that
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48 397 could be present in the ashes collected in a combustion power plant was estimated by
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1
2 398 recalculating the content of trace elements from biomass to ash by means of the following
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4 399 equation: $\%ELEMENT\ ash = \%ELEMENT\ biofuel / ASH\ CONTENT * 100$
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6 400
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9 401 It is important to note that when using this method, the levels are over-estimated because it
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11 402 does not take into account the losses derived from the volatilisation of these elements that
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13
14 403 would occur during combustion, particularly for elements with a very high volatilisation
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16 404 potential such as Hg and Cd. The levels obtained (Table 7) are well below the limits
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18 405 established in the European Directive 86/278/EEC that regulates the heavy metals of sewage
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20
21 406 sludge intended for soil application on agricultural land within the European Union, and thus
22
23 407 the ashes generated from the combustion of these short rotation crops could in principle be
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26 408 used as crop fertilisers in agriculture.
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31 410 However, several European countries have introduced requirements that are more stringent
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33 411 than this directive. For example, the levels of some elements in the ashes obtained from the
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35
36 412 studied biomass could exceed the limits established in Dutch regulations [42], the most
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38 413 restrictive national legislation in Europe. Further research is needed regarding the agricultural
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40
41 414 use of the different fractions of biomass ashes, as they are known to greatly differ in their
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43 415 particle size and chemical composition. In this sense, there is also a need to adapt regulations
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45 416 to the specific soil and climate conditions of the different regions.
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52 **Table 7:** Maximum levels of trace elements expected in the ash obtained after the combustion
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54 of the studied woody crops, and permissible limits of toxic elements for land application of
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sewage sludge. Units in mg/kg, dry basis

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2 422 The ash fusibility test indicated (results not shown) that all the species considered presented a
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4 423 good ash melting behaviour, with deformation temperatures higher than 1450 °C for most
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6 424 samples.
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11 426 It is not possible to compare the deformation temperatures since they are greater than the
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14 427 maximum values the available instruments can measure (1450 °C). To overcome this
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16 428 limitation, the relation between alkaline earth oxides and alkaline oxides (R) was used to
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18 429 predict the ash melting behaviour [33]. The alkaline-earth to alkali oxide ratio also suggested
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20
21 430 that all the studied species showed a similar ash sintering behaviour (Figure 8). This index
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23 431 improved during the third rotation cycle, in connection with the increase in Ca that was also
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25
26 432 noticed in the biomass produced during this cycle. In any case, this index was always
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28 433 maintained at levels higher than 2, and therefore the produced biomass is not expected to
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31 434 present any risk of sintering at normal boiler operating temperatures [33].
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34
35 436 **Figure 8:** Alkaline-earth to alkali oxide ratio (mean and LSD intervals) across species (left)
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38 437 and rotation cycles (right).
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4 CONCLUSIONS

The studied short rotation woody crops presented notably diverse results across species with regard to their biomass production and chemical composition, which could affect the combustion behaviour of the fuels produced from this biomass and their dedicated use.

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2 447 Willow, poplar and black locust produced 12, 9 and 7 dry Mg ha⁻¹ yr⁻¹ on average over the
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4 448 three rotation cycles established over 9 years, indicating that these species can be well-
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6 449 adapted to the unfavourable conditions of poor soils in low productivity areas in the
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9 450 Mediterranean region. In contrast, sycamore trees was not well adapted to the particular
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11 451 climate and soil conditions and provided extremely low yields, not even reaching 3 dry t ha⁻¹
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13 452 yr⁻¹ (9-year average), which suggests that the production of this biomass for dedicated energy
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15 453 crops might not be adequate for some Mediterranean areas, in particular marginal land with a
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18 454 continental influence.
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23 456 Biomass production sharply decreased during the third rotation cycle, suggesting the end of
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25 457 its productive cycle. Biomass produced in the third rotation cycle showed higher ash, Ca and
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27 458 calorific values than those observed during the previous rotation cycles, which was connected
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29 459 to higher bark-to-wood ratios as a consequence of the proliferation of small branches and
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31 460 twigs. Although significant, these increases were not found to affect the combustion
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33 461 properties of this biomass to any remarkable extent.
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41 Based on the ash chemical composition, the predictions of the alkaline earth to alkaline oxide
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43 464 ratio and the results obtained in the ash fusibility test, the biomass produced in the short
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45 465 rotation woody crops studied is not expected to pose sintering problems at normal combustion
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47 466 temperatures. Moreover, the low levels of heavy metals and other trace elements measured in
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49 467 the biomass produced suggest that, in principle, the ashes generated in the combustion of
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51 468 these woody short rotation crops could be applied to agricultural soils as crop fertilisers.
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60 471 Results suggest that poplar and willow biomass from dedicated energy crops in marginal land
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2 472 residential applications or wood pellets, class I3, for industrial applications. However, black
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4 473 locust biomass, a leguminous species with a high N-fixation capacity, would not be an
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6 474 appropriate raw material to produce industrial pellets because its N content could exceed the
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9 475 limits established in standard ISO 17225-2. However, the biomass produced from black locust
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11 476 energy crops could be used to produce wood chips, class B1.

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16 478 This study highlights the importance of taking into account not only biomass production but
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18 479 also the chemical composition of the biomass produced from dedicated energy crops, as it can
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21 480 highly affect the combustion properties of the fuel and its future use. It provides long-term
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23 481 information about biomass production and the combustion properties of the biomass produced
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26 482 in marginal land, which could be highly valuable when choosing woody species to produce
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28 483 biomass from energy crops for combustion purposes in the low productivity areas of the
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31 484 Mediterranean region. A future study of how the application of different soil enrichment
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33 485 techniques affects long-term biomass production and combustion properties of biomass
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36 486 produced on poor soil could also be very useful to increase the energy and economic value of
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38 487 short rotation woody crops.

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Table 1: Weather conditions measured (annual and global mean values) and at the experimental site.

Year	AMT (°C)	AmT (°C)	MMT (°C)	MmT (°C)	MT (°C)	P (mm)	Solar radiation (Wh m ⁻²)	Relative humidity (%)
2009	34.7	-14.0	17.7	4.4	10.8	389.8	1.6	64.8
2010	33.4	-11.0	15.6	4.2	9.6	598.5	1.5	68.0
2011	35.7	-11.9	17.9	4.9	11.1	379.6	1.6	66.5
2012	37.0	-10.3	17.7	4.5	10.6	344.4	1.7	62.9
2013	33.8	-8.8	16.5	3.2	9.5	595.0	1.6	70.4
2014	33.3	-6.7	18.0	3.6	10.7	596.0	1.6	68.7
2015	36.0	-9.5	18.1	4.6	11.0	488.1	1.6	67.1
2016	34.7	-7.5	17.3	4.8	10.7	540.2	1.4	69.5
2017	35.3	-11.7	18.5	4.8	11.9	314.8	1.7	63.0
Mean	34.9	-10.2	17.5	4.3	10.7	471.8	1.6	66.8
C.V.	3.6	23	5.1	13	7.0	25	5.1	4.1

AMT: Absolute maximum temperature, AmT: Absolute minimum temperature, MMT: Mean maximum temperature, MmT: Mean minimum temperature, MT: Mean temperature, P: Precipitation. , C.V.: Coefficient of variation.

Table 2: Soil characteristics at the experimental site.

Parameter	Horizon	
	0-30 cm	30-60 cm
Clay (%)	8.1	8.4
Silt (%)	5.9	6.6
Sand (%)	86	85
Soil texture	sandy	sandy
Gravels (%)	35	39
Density (Mg m ⁻³)	1.4	1.4
pH	6.8	6.5
Organic matter (%)	0.6	0.4
Carbonates (%)	1.9	< 0.1
N _{Olsen} (%)	0.03	0.03
P _{av} (mg kg ⁻¹)	6.7	7.4
K _{av} (mg kg ⁻¹)	61	66

av: available

Table 3: Standards and analytical methods of the properties measured in the woody SRC.

Property	Analytical technique	Standard
Sample preparation	Homogenisation, subsampling, milling	ISO 14780:2017
Moisture	Drying at 105 °C	ISO 18134-2:2015
Ash	Calcination at 550 °C	ISO 18122:2015
Volatile matter	Heating at 900 °C	ISO 18123:2015
C, H, N	Elemental analysis: IR and TCD (Leco)	ISO 16948:2015
S and Cl	Combustion bomb (Ika) + IC (Methrom)	ISO 16994:2016
Calorific value	Calorimetry (Ika)	ISO 18125:2017
Major elements	MW digestion (Milestone) + ICP-OES (Thermo Fisher)	ISO 16967:2015
Minor elements	MW digestion (Milestone) + ICP-MS (Thermo Fisher) For Hg: Gold amalgamation + AAS (Milestone)	ISO 16968:2015
Ash fusibility	Optical heating microscopy (Hesse)	CEN/TS 15370-1

Table 4: Proximate and ultimate analysis of the biomass across species and rotation cycles, and significance levels from ANOVA.

Species	Rotation cycle	Moisture (% wb)	Ash (% db)	Carbon (% db)	Hydrogen (% db)	Nitrogen (% db)	Sulphur (% db)	Chlorine (% db)	GCV (MJ/kg db)	NCV (MJ/kg db)
Black locust	1 st	29.4	2.1	48.6	6.2	0.84	0.05	0.02	19.55	18.21
Black locust	2 nd	37.2	2.2	48.8	6.1	1.00	0.05	0.01	19.64	18.31
Black locust	3 rd	30.5	2.1	49.3	6.2	0.76	0.04	0.02	19.77	18.42
	Average	32.4	2.2	48.9	6.2	0.86	0.05	0.02	19.65	18.31
	rsd (%)	13	3.2	0.74	0.83	14	18	33	0.55	0.56
Poplar	1 st	45.8	2.4	49.3	6.1	0.44	0.03	0.01	19.71	18.39
Poplar	2 nd	53.0	2.2	49.1	6.0	0.48	0.04	0.01	19.64	18.34
Poplar	3 rd	43.6	3.1	49.6	6.0	0.44	0.03	< 0.01	19.69	18.38
	Average	47.5	2.6	49.3	6.0	0.45	0.03	nd	19.68	18.37
	rsd (%)	10	18	0.51	0.85	5.7	10	nd	0.20	0.16
Sycamore	1 st	43.9	2.3	48.8	6.0	0.54	0.05	0.01	19.48	18.17
Sycamore	2 nd	45.8	2.2	48.6	6.0	0.54	0.06	0.01	19.45	18.15
Sycamore	3 rd	40.6	2.4	49.2	6.0	0.51	0.04	0.02	19.47	18.17
	Average	43.4	2.3	48.9	6.0	0.53	0.05	0.01	19.47	18.16
	rsd (%)	6.1	3.6	0.69	0.32	3.1	18	50	0.06	0.06
Willow	1 st	52.2	2.1	48.8	6.1	0.36	0.03	0.01	19.44	18.11
Willow	2 nd	53.9	2.3	48.6	6.0	0.47	0.04	0.01	19.48	18.17
Willow	3 rd	45.3	2.7	49.8	6.0	0.46	0.03	0.01	19.68	18.37
	Average	50.5	2.4	49.1	6.0	0.43	0.04	0.01	19.53	18.22
	rsd (%)	9.0	12	1.3	0.64	13	11	22	0.65	0.74
Global average		43.4	2.4	49.1	6.0	0.57	0.04	nd	19.58	18.27
Min.		29.4	2.1	48.6	6.0	0.36	0.03	< 0.01	19.44	18.11
Max.		54	3.1	49.8	6.2	1.00	0.06	0.02	19.77	18.42
Significance levels from ANOVA										
Species		nd	0.013	0.001	< 0.001	< 0.001	< 0.001	nd	< 0.001	0.001
Rotation cycle		nd	0.010	< 0.001	0.001	0.075	0.002	nd	0.015	0.014
Species-Rotation		nd	0.013	0.009	0.001	0.114	0.316	nd	0.150	0.143

wb: wet basis; db: dry basis; GCV: gross calorific value at constant volume; NCV: net calorific value at constant pressure; nd: not determined.

Table 5: Chemical composition of the biomass across species and rotation cycles, and significance levels from ANOVA.

Species	Rotation cycle	Al (% db)	Ca (% db)	Fe (% db)	K (% db)	Mg (% db)	Mn (% db)	Na (% db)	P (% db)	Si (% db)	Ti (% db)	Zn (% db)
Black locust	1 st	0.0022	0.59	0.0045	0.24	0.037	0.0018	0.0056	0.064	0.023	< 0.0002	0.0013
Black locust	2 nd	0.0012	0.60	0.0065	0.29	0.063	0.0025	0.0051	0.065	0.027	< 0.0002	0.0034
Black locust	3 rd	0.0029	0.59	0.0025	0.23	0.053	0.0017	0.0020	0.059	0.020	0.0003	0.0023
	Average rsd (%)	0.0021	0.59	0.0045	0.25	0.051	0.0020	0.0042	0.062	0.023	nd	0.0023
		41	1.3	44	13	25	21	46	5.0	15	nd	43
Poplar	1 st	0.0020	0.72	0.0038	0.31	0.076	0.0065	0.0044	0.079	0.027	< 0.0002	0.0042
Poplar	2 nd	0.0012	0.60	0.0065	0.29	0.063	0.0025	0.0051	0.065	0.027	< 0.0002	0.0034
Poplar	3 rd	0.0044	0.88	0.0038	0.34	0.078	0.0025	0.0030	0.088	0.029	0.0004	0.0034
	Average rsd (%)	0.0025	0.73	0.0047	0.31	0.072	0.0038	0.0042	0.077	0.028	nd	0.0037
		65	19	33	8.8	12	61	26	15	5.3	nd	12
Sycamore	1 st	0.0025	0.66	0.0045	0.29	0.090	0.0125	0.0036	0.102	0.030	< 0.0002	0.0014
Sycamore	2 nd	0.0012	0.60	0.0065	0.29	0.063	0.0025	0.0051	0.065	0.027	< 0.0002	0.0034
Sycamore	3 rd	0.0034	0.67	0.0029	0.26	0.060	0.0019	0.0023	0.067	0.023	0.0003	0.0026
	Average rsd (%)	0.0023	0.64	0.0046	0.28	0.071	0.0056	0.0037	0.078	0.026	nd	0.0025
		46	5.6	39	5.8	24	106	39	27	13	nd	40
Willow	1 st	0.0009	0.62	0.0034	0.23	0.056	0.0043	0.0039	0.066	0.015	< 0.0002	0.0027
Willow	2 nd	0.0012	0.62	0.0067	0.30	0.064	0.0025	0.0053	0.067	0.028	< 0.0002	0.0035
Willow	3 rd	0.0038	0.76	0.0032	0.30	0.068	0.0022	0.0026	0.076	0.025	0.0004	0.0030
	Average rsd (%)	0.0020	0.67	0.0044	0.28	0.063	0.0030	0.0039	0.069	0.023	nd	0.0030
		79	12	44	14	9.1	39	34	7.7	28	nd	13
Global average		0.0022	0.66	0.0046	0.28	0.064	0.0036	0.0040	0.072	0.025	nd	0.0029
Min.		0.0009	0.59	0.0025	0.23	0.037	0.0017	0.0020	0.059	0.015	< 0.0002	0.0013
Max.		0.0044	0.88	0.0067	0.34	0.090	0.0125	0.0056	0.102	0.030	0.0004	0.0042
Significance levels from ANOVA												
Species		0.126	0.004	0.761	0.009	0.003	0.013	0.565	0.024	0.031	nd	0.001
Rotation cycle		< 0.001	0.006	< 0.001	0.255	0.903	< 0.001	< 0.001	0.040	0.224	nd	0.001
Species-Rotation		< 0.001	0.031	0.135	0.005	< 0.001	< 0.001	0.118	< 0.001	< 0.001	nd	< 0.001

db: dry basis; nd: not determined.

Table 6: Trace elements of the biomass obtained from the short rotation woody crops during the third rotation cycle, and ISO 17225 requirements, parts 2 (woody pellets) and 4 (woody chips).

Species	As (µg/g db)	Cd (µg/g db)	Cr (µg/g db)	Cu (µg/g db)	Hg (µg/g db)	Ni (µg/g db)	Pb (µg/g db)
Black locust	< 0.2	< 0.1	< 1.0	2.9	0.002	3.6	< 1.0
Poplar	< 0.2	0.2	< 1.0	2.4	0.002	< 1.0	< 1.0
Sycamore	< 0.2	0.1	< 1.0	4.3	0.001	3.5	< 1.0
Willow	< 0.2	0.2	< 1.0	2.5	0.002	< 1.0	< 1.0
ISO 17225-2	≤ 1	≤ 0.5	≤ 10	≤ 10	≤ 0.1	≤ 10	≤ 10
ISO 17225-4	≤ 1	≤ 2	≤ 10	≤ 10	≤ 0.1	≤ 10	≤ 10

Table 7: Maximum levels of trace elements expected in the ash obtained after the combustion of the studied woody crops, and permissible limits of toxic elements for land application of sewage sludge. Units in mg/kg, dry basis.

Species	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn	Mn
Black locust	< 9	< 5	< 46	135	0.09	167	< 46	1081	918
Poplar	< 8	8	< 38	92	0.08	< 38	< 38	1406	1474
Sycamore	< 9	4	< 43	185	0.04	151	< 43	1061	2422
Willow	< 8	8	< 42	105	0.08	< 42	< 42	1274	1265
86/278/EEC limits	nl	20-40	100-150	1000-1750	16-25	300-400	750-1200	2500-4000	nl
Dutch limits	15	1.25	75	75	0.75	30	100	300	nl

nl: not limited

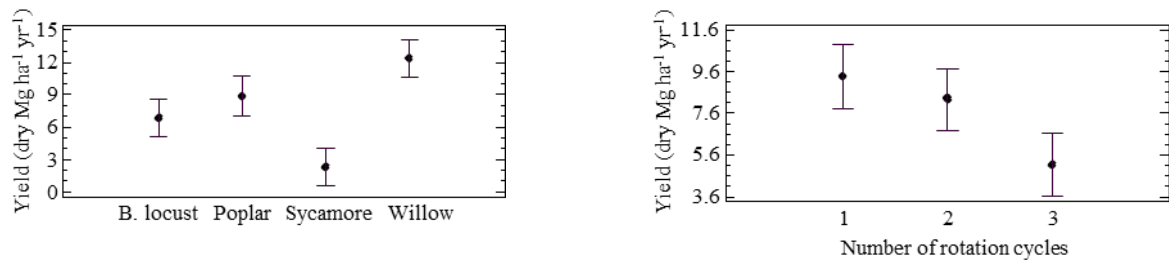


Figure 2: Dried biomass yield (means and LSD intervals) across species (left) and rotation cycles (right).

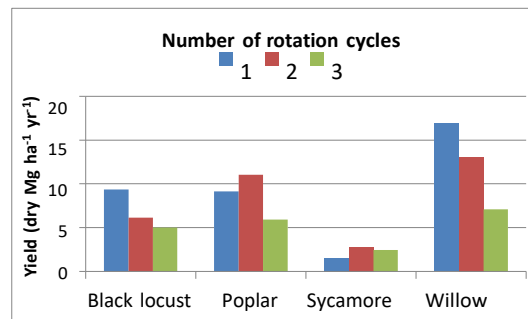


Figure 3: Dried biomass yield (Mg ha⁻¹ yr⁻¹) across species and rotation cycles.

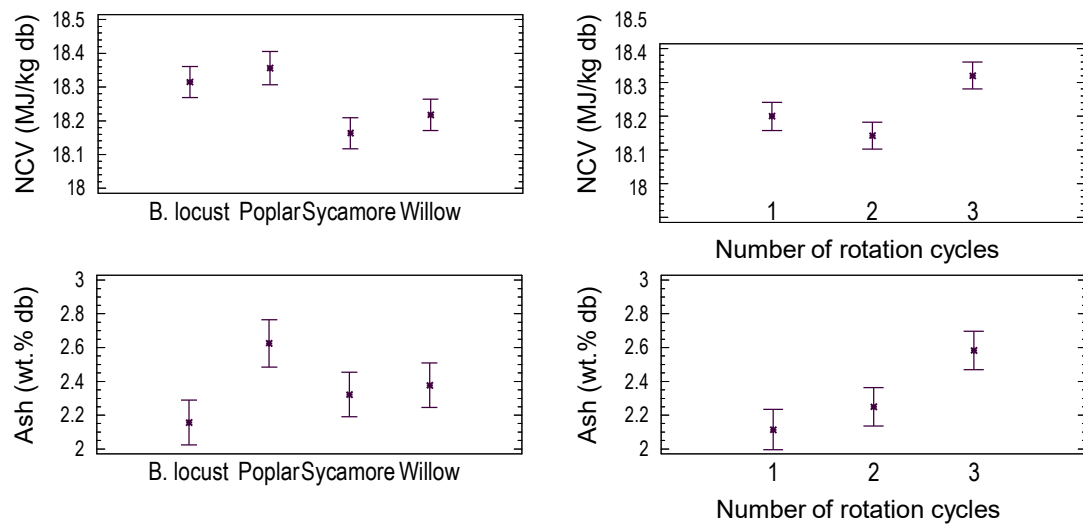


Figure 4: Net calorific value and ash content (means and LSD intervals) across species (left) and rotation cycles (right).

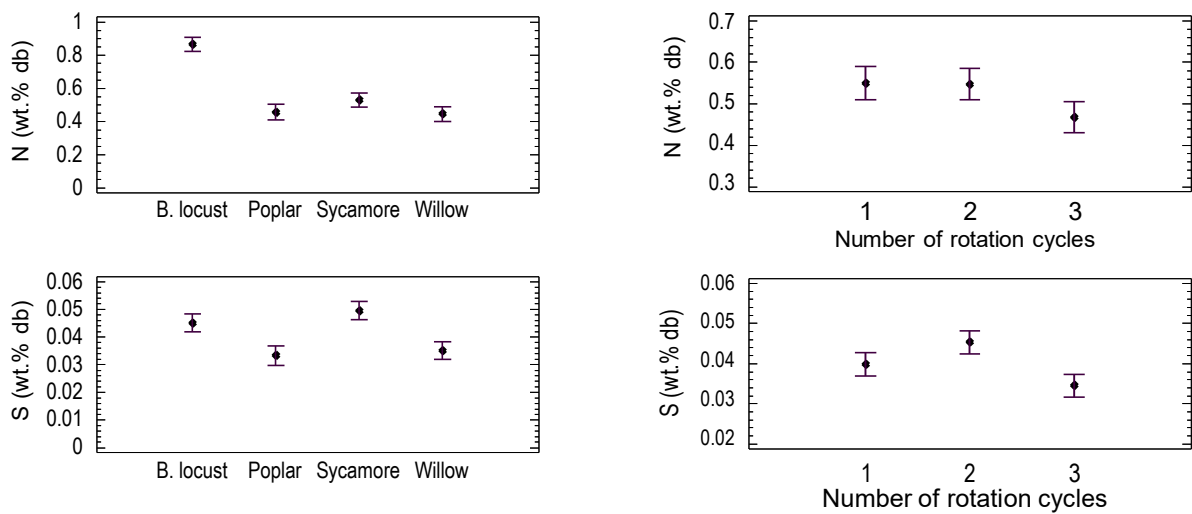


Figure 5: Nitrogen and sulphur contents (means and LSD intervals) depending on species (left) and rotation cycle (right).

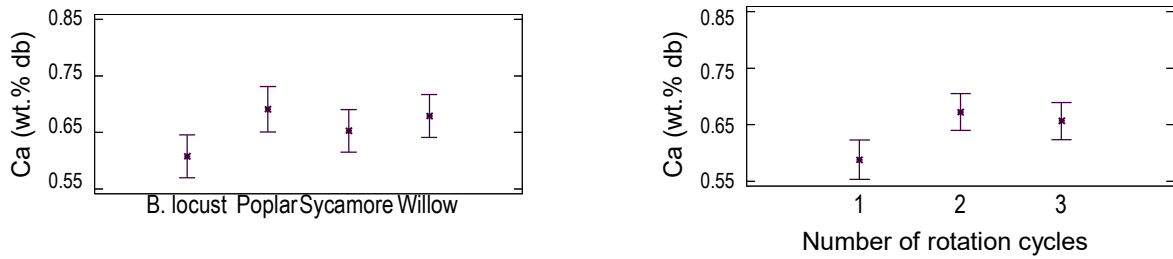


Figure 6: Calcium content (means and LSD intervals) depending on species (left) and rotation cycle (right).

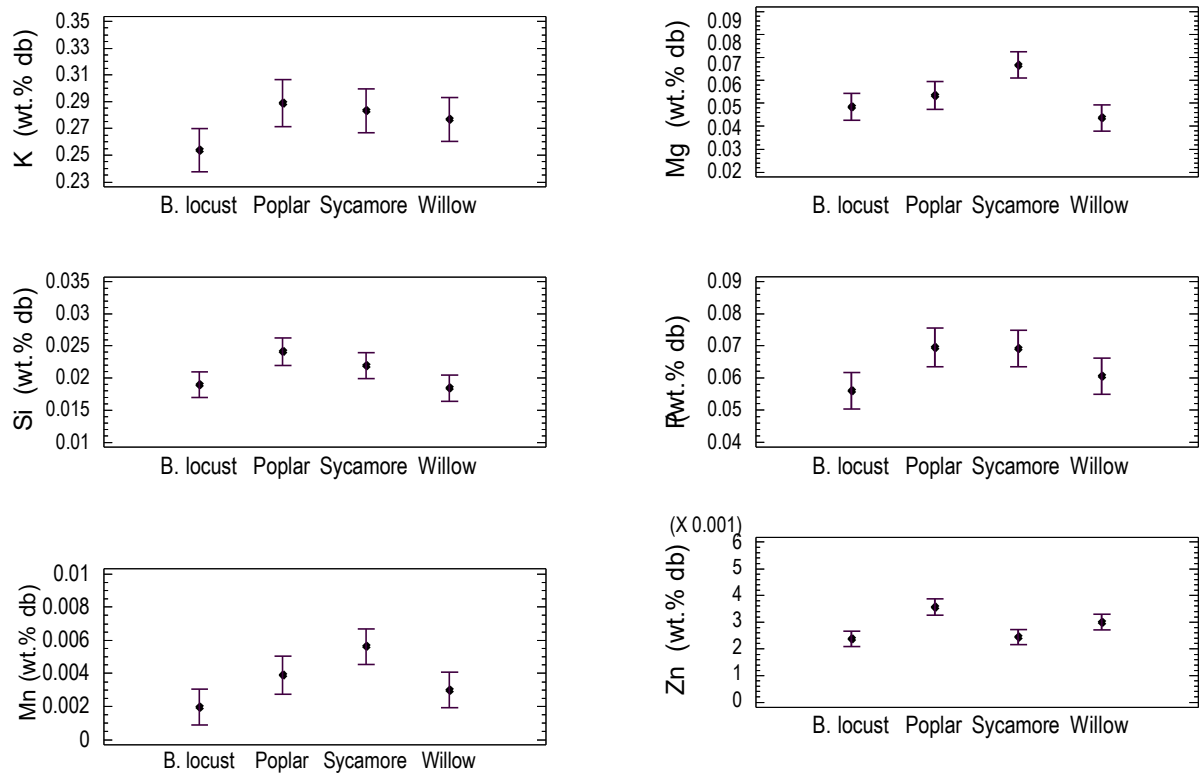


Figure 7: K, Mg, Si, Mn, P, and Zn contents (means and LSD intervals) depending on species.

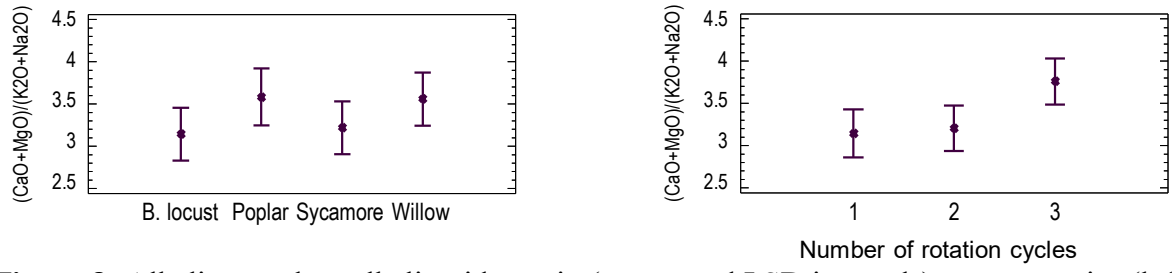


Figure 8: Alkaline-earth to alkali oxides ratio (means and LSD intervals) across species (left) and rotation cycles (right).

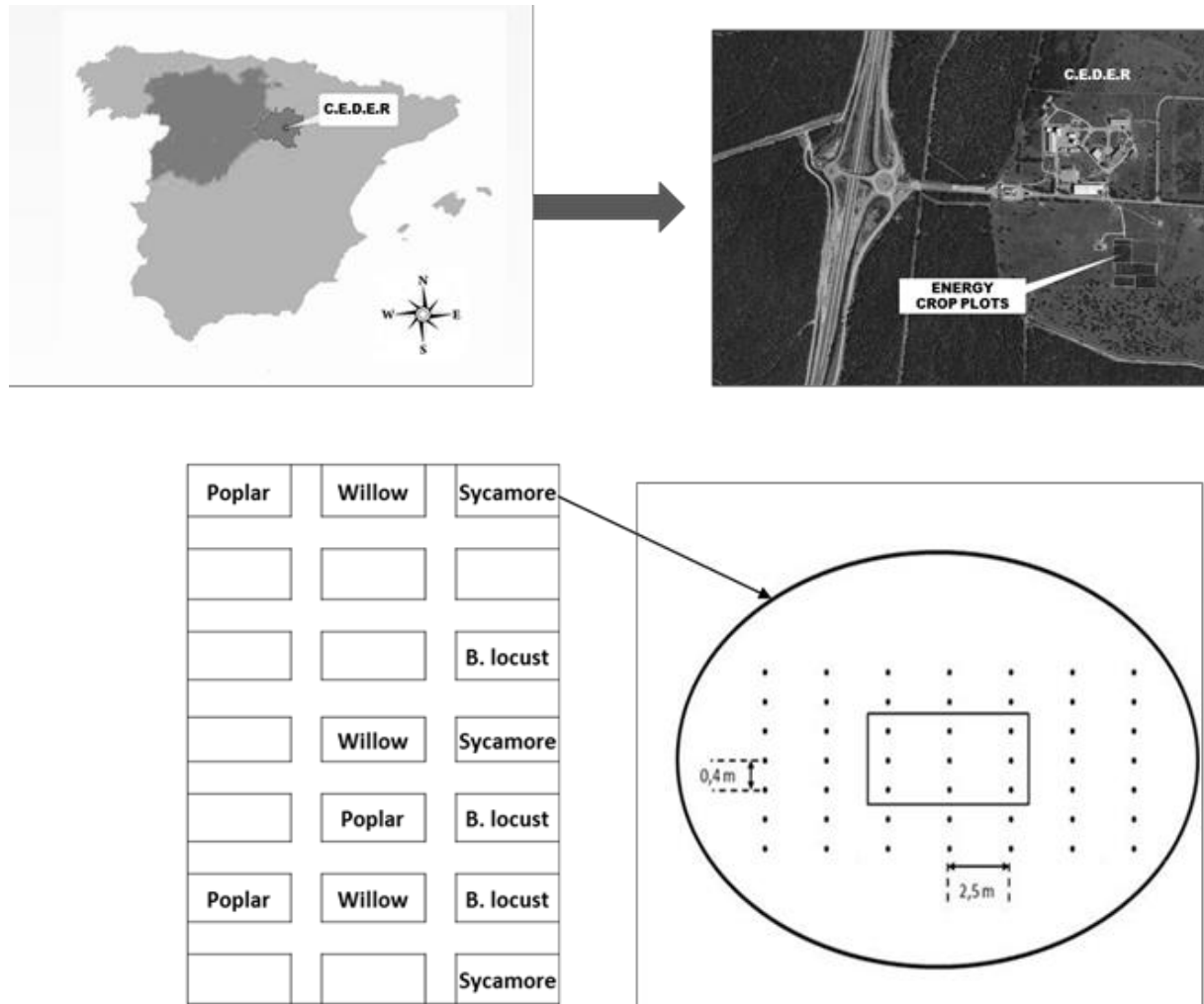


Figure 1: Location and distribution of species and trees at the experimental plantation. Each black dot represents an individual tree.

Interactive Map file (.kml or .kmz)



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