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Wood ash content variation in *Eucalyptus grandis* clones in Mozambique

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Highlights

- *Eucalyptus grandis* heartwood produces better fuel than sapwood, if assuming ash content as energy quality parameter.
- Younger individuals have higher ash content in sapwood, and older individuals in the heartwood.
- There was not significant stem end variation of ash content in heartwood and sapwood.

Abstact

The sustainability of native forests in Sub-Saharan Africa depends on the diversification of sources to generate bioenergy, and *Eucalyptus* spp. wood has been highlighted. However, the determination of energy quality parameters has been a challenge to enable plantation wood to generate energy. The research assessed the ash content of radial and longitudinal samples of *Eucalyptus grandis* (Hill) clone with different ages and growth sites. Samples were collected in three pre-established plots in the center of Mozambique. Five trees were cut down in each plot and six discs were removed from each tree. Grinded samples with <0.5 mm particle size were generated from the heartwood and sapwood of each disk to determine the ash content. Wood from 7-year-olds had a higher ash content compared to 9-year-olds. The two sample plots differed from each other in terms of wood ash content. Heartwood samples had smaller ash content than sapwood samples. In general, the ash content of the intermediate positions was lower than those from the base and top of the stem, for both radial sections. No conclusive differences were found between samples from the base and the top of the trees, indicating that the material from the top of the trees can also be used as wood fuel. Ash content can be a considerable parameter to assess the quality of the wood of *Eucalyptus* spp. as a fuel.

Keywords base-top; bioenergy quality; heartwood; sapwood; woody fuel
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1 Introduction

Worldwide, firewood, through direct consumption, and charcoal are the main ways of using energy from forests (Fundo Nacional de Energia 2014; Monjane and De Barros 2015). Wood fuels production makes a significant contribution to the energy needs of the African continent. Sub-Saharan Africa produces around 600 million m^3 of wood fuel per year, which covers 60–80% of energy needs, depending on the country. Mozambique is in fifth position with 4% of the ten largest charcoal producers globally (Steierer 2011). It should be noted that this value falls on natural forest, including the harvest of species protected by local law (Nhancale 2008; Afonso 2012; Atanassov-Andrade et al. 2012).

Although Mozambique is one of the few countries in Southern Africa that still has a considerable area of natural forests (Sitoe et al. 2012), the deforestation rate and forest degradation continues to increase, from 0.58% in 2007 to 0.77% in 2018 (Direcção Nacional de Florestas 2018). The harvest for energy production is one of the main drivers of deforestation (Cuamba et al. 2016; Egas et al. 2016). In 2008, annual consumption of wood fuels was estimated at 16 million m^3 $year^{-1}$. Ten years later, the volume of wood extracted from natural forests has increased to 20 million m^3 $year^{-1}$ (Nhancale 2008; Bila 2018). It is estimated that 95% of deforestation is caused by firewood and charcoal, which supply the energy needs of communities, bakeries, tea and tobacco drying (Bila 2018). Thus, over the years there has been no significant evolution regarding the dependence of the urban and rural population on the use of wood fuels in Mozambique (Ministério da Energia 2012, 2013). However, the availability of natural forests with the potential to continue to supply energy demand tends to decrease (Atanassov et al. 2012; DNF 2018) due to population growth, especially in rural areas, where about 85% of the population depends on biomass from forests for cooking, heating and as a source of income (Nhancale 2008).

As a way of guaranteeing the perpetuation of natural forests, environmental policies have been increasingly directed towards the protection of native forests. This emerges the need to use raw materials from reforestation (Varma and Behera 2009; Republic of Mozambique 2016). *Eucalyptus* spp. are the most widely planted exotic genus (Souza et al. 2004). Its rapid volumetric growth is pointed out as one of the important characteristics for energy generation (Goulart et al. 2003; Castro et al. 2013). However, wood is a heterogeneous and anisotropic material, with variations in its composition depending on the provenance, age of the trees, among other factors (Goulart et al. 2003; Castro et al. 2013), which alter the wood's energy qualifiers and quantifiers.

In the use of wood as fuel, physical properties such as wood density and moisture content; energetic properties (heating value); and chemical characteristics such as volatile material content, fixed carbon and ash content are important specific factors to be taken into account (Afonso 2012; Carneiro et al. 2014; Simetti et al. 2015).

Ash content is one of the most important parameters to evaluate the energy potential of wood. Ash originates from inorganic materials (silicone, aluminum, iron, calcium, magnesium, sodium, potassium, titanium and manganese) and does not participate in energy production (Frederico 2009; Santos 2010; Afonso 2012; Kajda-Szcześniak 2014). As it is an inorganic material, ash corresponds to the residues of the complete wood combustion. There is an inverse correlation between ash content, volatile materials content and heating value, and a positive correlation between heating value and fixed carbon content (Vale et al. 2002). Higher heating value is generally associated with material with higher levels of fixed carbon (Frederico 2009; Santos 2010; Castro 2011). On the other hand, high ash content in wood increase cleaning frequency and reduce the useful life of combustion equipment (Oliveira et al. 2010). Ash content also contributes to the particles emission through boiler chimneys, making it necessary to install separator particles equipment from combustion gases (Hytönen and Nurmi 2015; Čubars and Poiša 2017). The increase of particles in boilers causes corrosion of combustion equipment, cracks and fissures formation (Santos 2010). In domestic consumption, high ash content reduces heat propagation and energy value.

Studies on the determination of ash content directly in wood are scarce. However, several studies have been carried out on the immediate chemical analysis of charcoal, including the determination of ash content in charcoal (Vale et al. 2002; Andrade 2009; Barros et al. 2009; Afonso 2012; Zanuncio et al. 2014). However, the quantification and qualification of energy parameters is not linear in wood due to anisotropy. The heterogeneity of wood is manifested through the variation of its anatomical characteristics, physical and mechanical properties, and chemical characteristics between-species and within-species, growing under different ecological conditions (Vidaurre et al. 2012, 2013; Braz et al. 2014).

The data on wood energy quality of exotic species planted in Mozambique would allow the identification of *Eucalyptus* spp. with better quality for energy generation, and to understand the provenance variation of firewood energy quality in two important regions for forest plantation to maximize its domestic and industrial use. In this way, the ash content becomes a strong parameter to assess the quality and potential of woody fuels (Pereira et al. 1997; Calonego et al. 2005; Castro 2011; Johnson 2012; Hytönen and Nurmi 2015).

Recognizing the importance of ash content in wood to quantify wood quality, and the relevance of understanding variations in ash content at different ages, tree positions and growing regions, the research evaluated the ash content of radial and longitudinal samples of *Eucalyptus grandis* (Hill) with ages of 7 and 9 years. The study sites were plantations in Bandula and Penhalonga, the two important sites of reforestation in Austral Africa, center region of Mozambique.

2 Material and methods

2.1 Sample collection sites

The samples were collected in the localities of Bandula (19°20´S, 33°10´E) and Penhalonga (18°51´S, 32°50´E) in Manica province. Manica is located in the center of Mozambique, Sub-Saharan Africa (Fig. 1). As the results may be subject to climate and soil variations, the values for soil characteristics, temperature and precipitation for Bandula and Penhalonga were compiled (Table 1).

2.2 Samples collection

Eucalyptus grandis wood, aged 7 and 9-years, were used. The samples were collected from three blocks pre-established by the IFLOMA Company (Industria Florestal de Moçambique). Five trees were felled randomly at each location. Four 50-mm thick discs were removed from each tree: at the base (0.30 m from the ground), at 25%, 50%, and 75% of the commercial height of the tree (Trugilho et al. 2005; Castro 2011; Pereira et al. 2012; Simetti et al. 2015). Commercial height was considered to be the height which minimum diameter with bark was 60 mm.

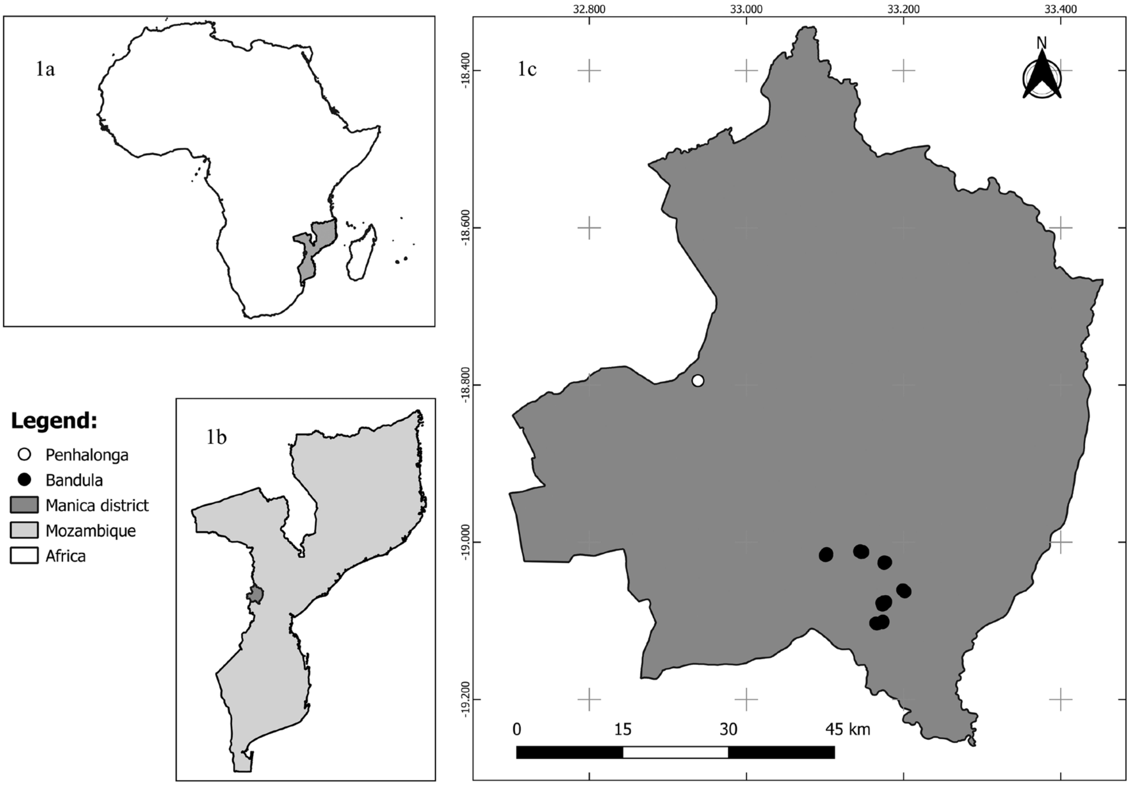

Fig. 1. Sites collection of *Eucalyptus grandis* discs samples. Africa (1a), Mozambique (1b), and Manica district boundaries highlighted (1c). The full black dots indicate discs' collection site in Bandula plantation and the white dot indicates Penhalonga plantation.

Table 1. Edaphic-climatic characteristics of the *Eucalyptus grandis* plantation sites. Data relating to geographic location, average temperatures, average cumulative amount of rainfall, elevation and soil typology of the sites.

Species	Age	Provenance	Temperature	Precipitation	Altitude	Soil depth	Soil type
Eucalyptus grandis	7 years	Bandula	24.06 °C	1138 mm	698 m	16.20 cm	Sandy clay franc
	7 years	Penhalonga	22.94 °C	1000 mm	813 m	16.00 cm	Sandy clay franc
	9 years	Penhalonga	22.94 °C	1000 mm	813 m	22.20 cm	Sandy clay

Sources: Instituto de Investigação Agronómica de Moçambique (1995) [Mozambique Agricultural Research Institute]; MAE (2005); Mina Alumina (2006); WorldClim (2020)

2.3 Samples preparation for laboratory analysis

The discs obtained from the different sections of the trees were sliced into four wedges according to the TAPPI T 605 om-92 (1992) standard. Two opposite wedges were used to determine the ash content. For each disk, four specimens (prisms) were produced with 20 × 20 × 50 mm dimensions (thickness × width × length), where the length corresponded to the thickness of the disk (Fig. 2). In each wedge, a prism was obtained in both heartwood and sapwood radial position. The prisms were reduced to chips. Chips originating from the five samples trees were mixed separated from each radial and axial position.

The samples were grinded using a Wiley-type knife mill into particles that passed through a 0.5 mm sieve. The particles were subjected to a Retsch sieve, and the sample that passed through the 0.5 mm sieve again, was used to determine the ash content, as described in the standard (ASTM D1102 – 84 2013).

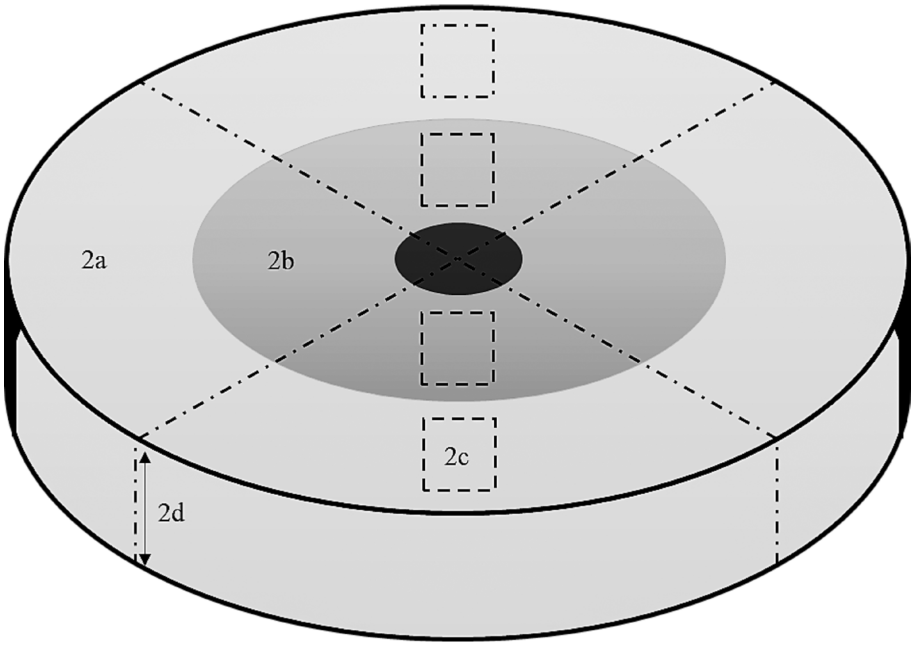

Fig. 2. Scheme of reduction of discs into laboratory test specimens. Sapwood (2a), heartwood (2b), transversal section of prism 20×20 mm (2c), and thickness of the disc (2d).

2.4 Assessment of ash content

The samples should be free of moisture. Two grams of the 0.5 mm diameter compound was weighed in 25 and 30 mL volume crucibles. The crucibles were previously heated in the Carbolite Gero muffle at a 600 °C temperature to stabilized mass. The samples in triplicate, and the respective crucibles were submitted in the oven at (103 ± 2) °C until the stabilization of the mass before being submitted to the muffle. The muffle was previously heated to approximately 100 °C, considered the initial temperature. The assessment of ash of wood followed the procedure described in the ASTM D1102 – 84 2013 standard. The effective time from the submission of the specimens to the total burning of the organic compounds in the wood and assessing ash was approximately 5 hours. The heating time of the samples in the muffle until the stabilization of the maximum temperature of the process (580 °C) was 1 hour and 10 minutes, with a heating rate of 7 °C min^{-1}. The ash was obtained after the complete combustion of the samples at 580 °C. The ash content was considered as the residual mass in relation to the anhydrous powder mass.

2.5 Statistical analyses

The ash content of wood was calculated separately for both heartwood and sapwood radial position, for each one of longitudinal section. The provenance, radial variance, and age mean were compared using Student's LSD test at a significance level $p < 0.05$. Longitudinal variance in both heartwood and sapwood, as well as age groups, were compared using ANOVA Tukey test at a significance level $p < 0.05$. The analysis was confined at four longitudinal positions: base (at 0.30 m of height from the ground), at 25%, 50% and 75% of commercial height. All analyses were carried out using the STATISTICA 8.0 program.

Table 2. Summary of the mean ash content of *Eucalyptus grandis* wood. The values in parentheses correspond to the variation coefficients. S1 indicates samples collected at 0.30 m height from the ground, and S2, S3 and S4 samples collected from 25%, 50%, and 75% of the commercial height of the tree, respectively. The data is grouped according to two test sites, two age groups, and two radial positions.

Site	Species	Age	Radial position	Mean ash wood content (%)			
				S1	S2	S3	S4
Bandula	*Eucalyptus grandis*	7-year	Sapwood	2.11 (3.00)	0.64 (22.31)	0.57 (6.43)	0.78 (4.95)
			Heartwood	0.33 (23.95)	0.33 (17.39)	0.36 (26.83)	0.34 (22.74)
Penhalonga	*Eucalyptus grandis*	7-year	Sapwood	0.65 (31.47)	0.49 (12.76)	0.59 (15.56)	0.46 (4.60)
			Heartwood	0.34 (0.03)	0.15 (51.78)	0.26 (11.67)	0.47 (2.97)
		9-year	Sapwood	0.52 (7.46)	0.35 (10.29)	0.31 (8.52)	0.34 (8.13)
			Heartwood	0.39 (5.69)	0.31 (9.34)	0.33 (4.51)	0.39 (15.14)

3 Results

Table 2 shows the mean ash contents and respective variation coefficients obtained for *Eucalyptus grandis* wood, according to age, provenance, radial and longitudinal position of the trees.

3.1 Age of the trees

Significant differences were observed between ash contents of 7 and 9-year-old *E. grandis* wood from Penhalonga plantations for both radial positions: heartwood ($t=3.52$; $P=0.02$) and sapwood ($t=3.49$; $P=0.03$).

3.2 Samples provenance

Analyzes were performed for section S2 only, at 25% of commercial height of 7-year-old samples. The wood from Bandula plantation showed higher ash content than the one from Penhalonga, both for heartwood and sapwood samples. The provenance had a significant effect on variation of ash content in heartwood samples ($t=3.38$; $P=0.03$). However, no differences in ash content were detected in the sapwood samples of *E. grandis* between Bandula and Penhalonga.

3.3 Sample position in the tree

3.3.1 Radial position

The ash content was higher in sapwood than in heartwood at all sampling sites and stem heights (Fig. 3). Significant differences were detected between heartwood and sapwood samples in all sections studied from 7-year-old trees, except at 75% section from Penhalonga samples. Differences between 9-year-old heartwood and sapwood samples were detected only for S1 from Penhalonga plantation ($t=1.37$; $P=0.01$).

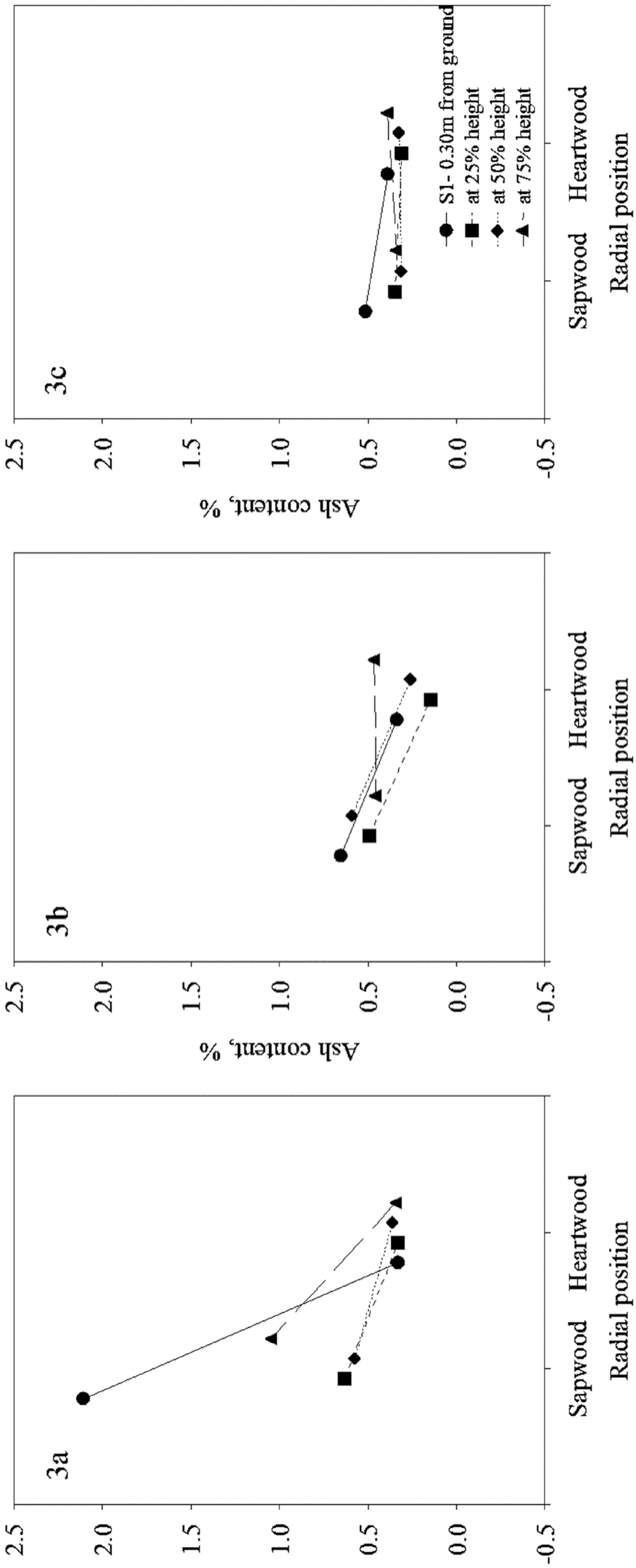

Fig. 3. Effect of radial position on ash content in 7 and 9-year-old *Eucalyptus grandis* wood. Radial variation of ash content of 7-years-old Bandula samples (3a), 7-year-old (3b), and 9-year-old (3c) both from Penhalonga samples; round symbol stands for the samples extracted at 0.30 m height from the ground, quadrangular symbol for the samples extracted at 25% height, lozenge symbol for the samples extracted at 50% height, and triangular symbol for samples extracted at 75% height, in relation to the commercial length of the stem.

3.3.2 Longitudinal position

Higher ash contents were found in samples from the base and the top of the trees, with the base showing higher contents. The lowest ash contents were found in intermediate samples (at S2 and S3) (Fig. 4). ANOVA found significant differences in ash content only in the *E. grandis* sapwood samples of 7-year-old collected in Bandula ($F = 214.48$; $P < 0.00$). However, the Tukey test did not find differences in the sapwood samples at 25% and 50% of commercial height from same plantation. For the 7-year-old samples from Penhalonga, only the heartwood samples were statistically different along the longitudinal axis ($F = 27.90$; $P < 0.00$). Tukey's test showed differences in mean ash content between S1 and S2 ($P = 0.01$), and S2 and S4 ($P < 0.00$). In 9-year-old individuals, ANOVA showed differences along the length of the trees only in the radial sapwood position ($F = 25.88$; $P < 0.00$). However, just sapwood samples extracted at 0.30 m from the ground differed from all other longitudinal positions.

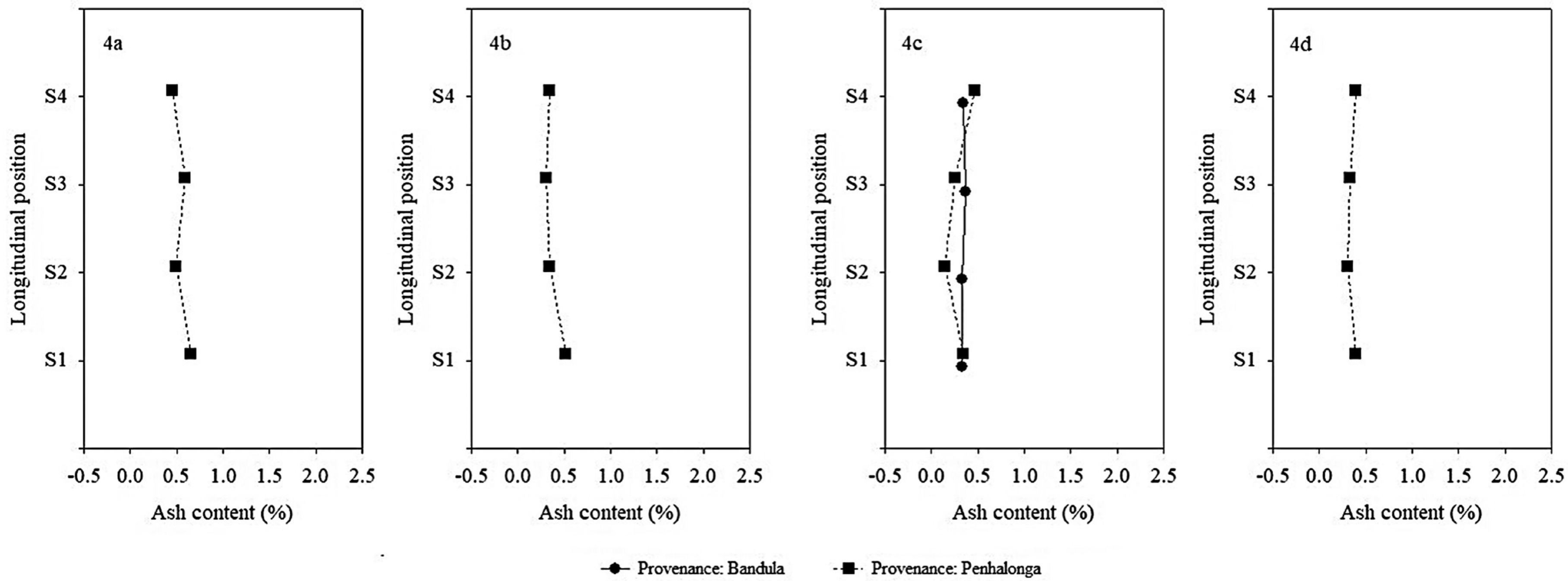

Fig. 4. Variation of ash content at different heights of 7 and 9-year old *Eucalyptus grandis* stems. 4a: 7-year-old sapwood samples; 4b: 9-year-old sapwood samples; 4c: 7-year-old heartwood samples; 4d: 9-year-old heartwood samples; round symbol stands for the Bandula site and rectangular symbol for the Penhalonga site.

4 Discussion

4.1 Effect of age on ash content variation

The results show that for sapwood, the ash content decreases with the age of the trees, which corroborates with the findings of several authors. Carvalho (1997) analyzing a hybrid *Eucalyptus grandis* × *E. urophylla* of ages 4, 7 and 9 years, Castro (2011) analyzing clones of *Eucalyptus urophylla* (S. T. Blake) and hybrids of *E. grandis* × *E. urophylla* with ages of 3, 4, 5 and 7 years, and Soares et al. (2015) studying the same hybrids with ages of 3, 5 and 7 years found a decrease in ash content with increasing age. Previously, ash content ranging from 0.70 to 0.22% was observed for *Eucalyptus saligna* (Smith) from 1 to 4-year-old, respectively (Trugilho et al. 1996). De Morais et al. (2017) found that a 1-year-old *E. grandis* × *E. urophylla* hybrid had a higher ash content than the 8-year-old did. On the other hand, the present study indicates that the ash content of core samples from 7-year-old individuals is lower than that of 9-year-old individuals. However, the difference in ash content between 7- and 9-year-old individuals is not conclusive, especially as the difference in heartwood and sapwood ash content in older trees is minimal (Table 2). Using the same origin samples of these study, Varela (2019: unpublished) did not find a significant difference in wood density for 7 and 9-year-old, both for the heartwood and sapwood radial position. That fact needs further research.

4.2 Effect of the provenance of the species

The influence of planting location on ash content was observed by some authors. The lowest ash content (0.73%) for a 3-year-old *E. grandis* clone was found in samples from Santa Bárbara and the highest in clones from other regions of Guanhães (0.99%) and (1.26%) in Ipaba, Brazil (Frederico 2009). Precipitation and altitude proved to be important for the amount of ash in the wood. A similar pattern was observed in the present study: trees from Penhalonga region, a mountainous area, had a lower ash content than trees from Bandula that is a plain region. However, both 7-year-old *E. grandis* plantation sites have similar soil types and depths (Table 1). Thus, the altitude had a greater contribution to low ash contents in Penhalonga than the precipitation, once the precipitation in Bandula region is 138 mm higher. However, other factors may be associated with the findings, too. For example, the change in the nutritional status of the plant is one of the factors that determine variations in the ash content of wood (Castro 2011; Castro et al. 2013; Hytönen et al. 2018). Analyzing *E. grandis* × *E. urophylla* hybrids from 1 and 8-year-old plantations, it was concluded that the change in plant metabolism was influenced by the characteristics of climate, soil, and topography of the site (Santos 2010; De Morais et al. 2017).

4.3 Effect of the sample radial position

In general, lower ash content was found in the region closest to the pith (heartwood) in relation to the region close to the bark (sapwood). Similar behavior was found in *Eucalyptus* spp. when determining high values of density and ash content as it advanced from the pith to the bark (Trugilho et al. 2005). Woods with high density generally have thick cell walls and accumulate more cellulosic material and minerals per unit volume as they grow (Ciolkosz 2010). Studying the density at the 1.3-metre-height of the same trees used in the present study, it was observed that sapwood's basic density was higher than that of heartwood (Varela 2019). There is a strong negative correlation between ash content and cell lumen diameter, indicating an increase in ash content for woods with small lumen and higher density (Santos 2010). Sapwood is the physiologically active part of wood, composed of living cells, where all types of compounds are transported before being stored in the heartwood (Pereira et al. 2013; Silveira et al. 2013). The ash content in samples from plantation with 9-year-old individuals was not significantly affected by the radial position, regardless of the longitudinal position. This fact may probably result from a decrease in the metabolic activity of sapwood with increasing age (Castro 2011; Castro et al. 2013).

4.4 Variation of ash content along the longitudinal axis

Studies of ash content variation in the radial (bark-to-pith) and longitudinal (bottom-to-top) directions are extremely important to determine the quality and location of extraction of wood for energy (Brito et al. 1983). These variations result from anatomical differences between heartwood and sapwood, as well as changes in the growth rings (Vale et al. 2009). The highest values of ash content were verified for the positions of the base and at 75% of the commercial height. In a similar study, the lowest ash contents were found at 25% height (0.15%) and 50% height (0.19%) in relation to the commercial length of the stem (Simetti et al. 2015). The authors observed that the ash content at 25% height was lower than at the other positions in the tree. In another study, lower ash contents were found at the 1.3-metre-height (0.50%) and at 50% height (0.42%), and higher at base (0.71%), 25% (0.74%), 75% (0.72) and at 100% height (0.65 %) in five 8-year-old *E. grandis* × *E. urophylla* clone hybrids, without, however, finding statistical differences in ash content between any of the positions of the sampled trees (Coelho et al. 2018). In the present research, a generalized trend of ash content along the longitudinal axis was not found. However, in most cases the intermediate sections S2 and S3 had lower ash content in relation to the other positions along the height of the trees. Similar trends were observed by (Simetti et al. 2015; Dibdiakova et al. 2017; Coelho et al. 2018). On the other hand, for all cases, the ash content decreases from the S1 section (base, at 0.30 m from the ground) to the S2 section, which indicates that the base wood has a lower quality for energy, if considered the ash content as a quality parameter, particularly considering the ash content of 7-year-old *E. grandis* collected in Bandula (2.11%).

5 Conclusions

Considering the ash content of *Eucalyptus grandis* wood as a quality indicator in energy production, the heartwood material produces better fuel than the sapwood material. Younger individuals have a higher content of ash in the sapwood than in the heartwood. Although the ash content of *E. grandis* is mostly within the admissible amounts for domestic or industrial use, samples from Penhalonga plantations have better energy quality than those from Bandula plantations. No conclusive differences were found between samples from the bottom and top of the trees. This means that the samples from the tops of the trees can also be used as wood fuel, yet the bottom and intermediate wood is commonly given supremacy. The ash content proved to be a considerable parameter to evaluate the quality of *Eucalyptus* spp. as a woody fuel in center region of Mozambique, as it allows the selection of the best age, ideal regions for growth and position of trees capable of providing wood with a lower ash content. However, the results of the analyzes showed the need to include other variables that can better explain the reasons and patterns of ash content variation in *Eucalyptus* spp. wood.

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Availability of research materials and data

The data and other research supplementary material required will be available for Silva Fennica, and other databases where Silva Fennica is listed when registering the DOI of the article.

Authors' contributions

The contributions of the authors are as follow:
Semo Mogeia: conception of research question and design of the work; acquisition, analysis and interpretation of the data and results; scientific writing; final approval of the version to be published; and accountable for all aspects of the work in ensuring that questions related to the accuracy are appropriately investigated and solved. **Alberto A. Manhiça** and **Andrade F. Egas**: The acquisition, analysis and interpretation of the data and results; revising it critically for sound and intellectual content; final approval of the version to be published; and integrity of any part of the work are appropriately investigated and solved. Andrade F. Egas also contributed as general supervision of the research group.

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