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An Analysis of Sampling Methods for Determining Calorific Energy Values Of Forestry Species

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ABSTRACT

Background: The evaluation of different methods and sample sizes is an issue of great importance when conducting studies with large sample sizes, as cost and time can become prohibitive. And using small sample sizes can lead to great errors in the estimation of the true population mean. This study aimed evaluate the most accurate combinations of samplings point along tree boles for the estimate of gross calorific value (GCV) for the species *Acacia mearnsii* De Wild, *Eucalyptus grandis* W. Hill ex Maiden, *Mimosa scabrella* Benth. And *Ateleia glazioveana* Baill. The blocks were characterized by a 4x5 factorial with four forest species (*Acacia mearnsii*, *Eucalyptus grandis*, *Mimosa scabrella* and *Ateleia glazioviana*) and measurements were taken along five positions on the bole, with three replications. Disks of about two centimeters were sampled along 0%, 25%, 50%, 75% and 100% of a given tree'sheight; the samples were then pulverized, and made into disks for analysis by a digital calorimeter. Pearson correlation analyses, and modeling via multiple stepwise regressions were carried out. Three year old trees of the species were arranged in a spacing arrangement of 3.0x1.5 m. The average values of GVC for the species *Acacia mearnsii*, *Mimosa scabrella*, *Eucalyptus grandis* and *Ateleia glazioviana* are 4.423, 4.511, 4.523 and 4.628 kcal kg^{-1}, respectively. For the analysis of composite samples, it is recommended that for, *Acacia mearnsii*, *Mimosa scabrella* and *Ateleia glazioveana*, groupings of samples should ideally be taken from the position of 0% (base height) and 25%, or 25% and 50%, or 50% and 75%. For *Eucalyptus grandis*, three disks at the position of 0%, 50%, and 100% of tree height must be sampled for accurate measurements.

INTRODUCTION

With the growing demand for renewable energy sources, studies of the potential trees for the generation of forest biomass have been increasingly conducted in Brazil and in the world. Many studies have demonstrated the potential of biomass for clean energy production, such as the projects developed by Lima *et al*. (2011); Vidaurre *et al*. (2012); Protásio *et al*. (2013); Caron *et al*. (2015) and Eloy *et al*. (2015). In order to increase wood conversion efficiency, the adoption of more appropriate technologies is needed. Such improvements would lead to more accuracy when assessing the true carbonization potentials of tree species, and when obtaining heat and in the generation of energy (Silva *et al*., 2012). Similarly, in planning the production of an area, a forestry company should be evaluated all aspects that may directly or indirectly influence final costs, as well as end-use of wood (Trevisan *et al*., 2016).

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In the case of biofuels, the gross calorific value (GCV) is regarded as the most important feature when assessing the quality of materials for energy purposes. According to Eloy *et al*. (2016), the GCV is the amount of energy in the form of heat released during the full firing of a particular unit of mass.

The rule of the 'Brazilian Association of Technical Standards' NBR 8633 (ABNT, 1984) defines the way in which one should perform the GCV analysis; however, it does not mention how to proceed with the sample timber of a tree. Thus, sampling methods used by several researchers have varied considerably, as can be seen in many works; there does not appear to be a standard in for this type of timber tree sampling.

Vale *et al*. (2000) studying de *Eucalyptus grandis* and *Acacia mangium* species, used a sample of five positions along the bole, 0%, 25%, 50%, 75% and 100% of the total height. Eloy *et al*. (2014), when comparing the energy production of native and exotic species in forest plantations, also used a sample of five positions along the bole, 0%, 25%, 50%, 75% and 100% of the total height. Pereira and Higa (2003), studying the properties of *Taxodium distichum* (L.) Rich. wood, used composite samples of disks taken every 2.80 m beginning from the bottom and ending at the top of tree. Silva *et al*. (2012), estimating the GCV at different sampling points along the bole, and recommended for *Acacia mearnsii*, *Mimosa scabrella* and *Eucalyptus grandis*, the measurement of disk samples in the positions 0%, 25% and 75% of total height for an accurate composite sample.

The differences reported in the literature must be refined. The costs and time involved when working with large samples is can be cost prohibitive, and when too small, lead to errors in estimation (Silva *et al*., 2012). Our objective in this study was to analyze sampling points for GCV (gross calorific values) along the bole of trees, for the species *Acacia mearnsii*, *Eucalyptus grandis*, *Mimosa scabrella* and *Ateleia glazioviana*.

MATERIAL AND METHODS

Study area and experimental design:

The study was conducted in experimental area of the Federal University of Santa Maria, campus Frederico Westphalen - RS, Brazil, with geographic coordinates 27° 23' 26" S; 53° 25' 43" W, 461m. According to the Köppen climate classification (Alvares *et al*., 2013), the climate of the region is Cfa, ie, humid subtropical with an average annual temperature of 19.1°C, varying with maximum of 38ºC and minimum of 0°C.

The experiment was evaluated using a randomized complete block design. The blocks were characterized by a factorial 4x5, ie, four forestry species (*Acacia mearnsii*, *Eucalyptus grandis*, *Mimosa scabrella* and *Ateleia glazioviana*) and five positions along the trunk (0%, 25%, 50%, 75% and 100% of the total height), with three repetitions. Each block was composed of four experimental units and different treatment was carried out in unit, with a total of 45 trees in each block. Four trees were collected per repetition in each treatment, totaling 12 trees per species.

Evaluation of the Gloss calorific value (GCV):

The collection of samples was performed in September of 2011, three years after planting. For the 48 trees submitted to the spacing of 2.0 x 1.5 m, wood samples were processed in the form of disks approximately two centimeters thick, along the points of 0%, 25%, 50%, 75% and 100% of total height.

To determine the GCV, discs were cut in a mill and passed through a 40 mesh sieve, in order to obtain finer and more uniform material. This was then taken to an air-circulation oven and heated until the samples reached a constant weight, in order to determine the mass of dry matter. After being withdrawn from the air-circulation oven, they were placed in a desiccator for cooling; subsequent GCV determinations were carried out with a digital calorimeter (C5000, IKA WORKS) according to NBR 8633 (ABNT, 1984).

In order to evaluate the quantity and ideal groupings of discs required for good sampling and minimize the possibility of error, it is recommended to sample along the bole for determining species' GCV. The multiple regression procedures among the averaged values of the individual samples of different sampling combinations of the disks along the bole can be seen in Table 1.

Table 1: Number of disks per sample combination along the bole (0%, 25%, 50%, 75% and 100%) in relation to the total height, for determining the gross calorific value of forestry species.

Number of disks by sampling combination			
1 disk	2 disks	3 disks	4 disks
0%	0+25%	0+25+50%	0+25+50+75%
25%	0+50%	0+25+75%	0+25+50+100%
50%	0+75%	0+25+100%	0+25+75+100%
75%	0+100%	0+50+75%	0+50+75+100%
100%	25+50%	0+50+100%	25+50+75+100%
-	25+75%	0+75+100%	-
-	25+100%	25+50+75%	-
-	50+75%	25+50+100%	-
-	50+100%	25+75+100%	-
-	75+100%	50+75+100%	-

Statistical analysis:

The GCV data were statistically analyzed using the software Statistical Analysis System (SAS, 2003), which was used to conduct an analysis of variance and F test; data normality and homogeneity of variances were verified.

For statistical analysis, independent variables were analyzed for the combinations of one, two, three, and four discs were used, resulting in a total of 5 combinations. Pearson correlation and multiple regression between the variables involved were analyzed. Multiple regression analysis was performed by the stepwise method, and the combination of disks selected for the regression model was considered the form best sampling, the possibilities of one, two, three, or four discs along the bole. These analyses were performed in the Software "Statistical Analysis System" (SAS, 2003), in the regression subprogram using the procedure Proc stepwise.

RESULTS AND DISCUSSION

Analysis of variance and gross calorific value:

From the analysis of variance, there was a significant difference for GCV, among the four forest species. This difference was not observed for GCV at different positions along the bole, nor for the interaction of position x species (Table 2). The GCV mean values were 4.423 kcal kg^{-1} for *Acacia mearnsii*; 4.511 kcal kg^{-1} for *Mimosa scabrella*; 4.523 kcal kg^{-1} for *Eucalyptus grandis* and 4.628 kcal kg^{-1} for *Ateleia glazioveana*.

Table 2: Analysis of variance for the gross calorific value of forest species at different positions along the bole in relation to the total height.

Factor study	Freedom degree	Mean square gross calorific value
Species	3	101348.9823*
Position	4	1278.0933^{ns}
Species x Position	12	3352.7233^{ns}
Block	2	3383.5202^{ns}
Determination coefficient		0.76
Coeffient of variation		1.21

Where: * = significant at 5% probability of error as the Fisher distribution; ^{ns} = not significant at 5% probability of error as the Fisher distribution.

GCV values were higher than reported in the literature. According to Silva *et al*. (2012), estimating the GCV along the bole of tree species, found for species *Eucalyptus grandis*, *Acacia mearnsii*, *Ateleia glazioveana* and *Mimosa scabrella* GCV of 4.346 kcal kg^{-1}, 4.482 kcal kg^{-1}, 4.492 kcal kg^{-1} and 4.511 kcal kg^{-1}, respectively.

Similarly, Eloy *et al*. (2014) reported GCV values for *Eucalyptus grandis* that ranged from 4.241 kcal kg^{-1} to 4653 kcal kg^{-1}, as well as, for *Mimosa scabrella* from 4.349 kcal kg^{-1} to 4.679 kcal kg^{-1}, *Acacia mearnsii* between 4.442 kcal kg^{-1} to 4.828 kcal kg^{-1}, for *Ateleia glazioviana* 4.440 kcal kg^{-1} to 4.586 kcal kg^{-1}, when evaluating trees for three years. Sturion and Tomaselli (1990) found the GCV of 4.414 kcal kg^{-1} for *Mimosa scabrella*, lower than the average found by Silva *et al*. (1982) and Silva and Biassio (2009), which was 4.700 kcal kg^{-1} and 4.782 kcal kg^{-1}, respectively.

The GCV value found in our study for *Eucalyptus grandis* (4.523 kcal kg^{-1}) is within the range reported for Quirino *et al*. (2005), which varied from 4.501 kcal kg^{-1} to 4701 kcal kg^{-1}, for the same species. Pereira *et al*. (2000), studying several species of *Eucalyptus*, observed values ranging from 4.340 kcal kg^{-1} for *Eucalyptus grandis* with three years of age, to 5.080 kcal kg^{-1} for *Eucalyptus camaldulensis* with six years of age. Jara (1989) and Vale *et al*. (2000) found a GCV of 4.790 kcal kg^{-1} and 4.641kcal kg^{-1}, respectively, for seven year old *Eucalyptus grandis* trees. For *Ateleia glazioveana*, Mattos *et al*. (2000) and Baggio (2002) reported GCV of 4.637 kcal kg^{-1} and 4.450 kcal kg^{-1}, respectively. Kannegiesser (1990), observed value of 3.744 kcal kg^{-1} for *Acacia mearnsii*.

All GCV values found in this study were within the range found by Quirino *et al*. (2005); these authors revised one hundred different tree Brazilian species and reported the average value of 4.732 kcal kg^{-1}, ranging from the lower limit of 3350 kcal kg^{-1} to the upper limit of 5260 kcal kg^{-1}.

Number and location of disks along the bole affected the estimation of gross calorific value:

We observed changes between the averages for each position, along the bole, especially when we have the use of one disk (0, 25, 50, 75 or 100%) (Figure 1). Some properties of the wood can influence the value of GCV; for example, the specific mass (Gatto *et al*., 2003), lignin content, flammable extractives (oils, resins, waxes) (Burger and Richter, 1991), and the chemical composition of the wood (carbon, hydrogen, nitrogen, oxygen and sulfur) (Munalula and Meincken, 2009).

According to the results obtained from estimating the GCV in relation the number of disks along the bole, the ideal groupings of plant height samples necessary for accurate estimates are as follows: *Acacia mearnsii*: one (75%), two (0% + 25%), three (0% + 25% + 100%) or four (0% + 25% + 50% + 75%) disks; if one were to sample *Mimosa scabrella*, the ideal grouping options are one (25%), two (25% + 50%), three (0% + 75% + 100%) or four (0% + 50% + 75% + 100%) disks.

Groupings for *Eucalyptus grandis* analyses are one (100%), three (0% + 50% + 100%), or four (0% + 25% + 50% + 75%) disks. For *Ateleia glazioveana* the most ideal groupings are two (50% + 75%) or three (0% + 25% + 100%) disks. This data is based upon the high determination coefficients (Table 3) and Pearson correlations (Tables 4 and 5) conducted in this study. One can reduce the costs of sampling, GCV analysis, and achieve good statistical values with this sampling regimen.

Table 3: Selection of sampling combinations along the stem, selection and testing of models to estimate the higher calorific value of the four forest species: *Acacia mearnsii*, *Mimosa scabrella*, *Eucalyptus grandis* e *Ateleia glazioveana*.

Selecting combinations sampling				
Species	Number of disks in combinations			
	1	2	3	4
Eucalyptus grandis	100%	ns	0+50+100%	0+25+50+75%
Acacia mearnsii	75%	0+25%	0+25+100%	0+25+50+75%
Mimosa scabrella	25%	25+50%	0+75+100%	0+50+75+100%
Ateleia glazioveana	ns	50+75%	0+25+100%	ns

Selection and testing of models		
	Combination / Model	R^2
Eucalyptus grandis	GCV1 = 1362.7101+0.7105*(100%)	0.986
	GCV2 = ns	-
	GCV3 = 2139.3889+0.5291*(0+50+100%)	0.975
	GCV4 = -495.7676+1.1051*(0+25+50+75%)	0.997
Acacia mearnsii	GCV1 = -40.7825+1.00875*(75%)	0.960
	GCV2 = 1357.7693+0.6921*(0+25%)	0.999
	GCV3 = -195.7166+1.0436*(0+25+100%)	0.999
	GCV4 = 895.1372+0.7974*(0+25+50+75%)	0.999
Mimosa scabrella	GCV1 = 578.9137+0.8739*(25%)	0.999
	GCV2= -866.9121+1.1914*(25+50%)	0.988
	GCV3 = 474.0123+0.8953*(0+75+100%)	0.997
	GCV4 = -171.8087+1.0374*(0+50+75+100%)	0.999
Ateleia glazioveana	GCV1 = ns	-
	GCV2 = 4243.9521+0.0829*(50+75%)	0.998
	GCV3 = 5353.5017+0.1568*(0+25+100%)	0.997
	GCV4 = ns	-

Where: 1 = average value of the individual samples of disks (0%, 25%, 50%, 75% and 100%); 2 = average values from the combination of two disks (0+25%, 0+50%, 0+75%, 0+100%, 25+50%, 25+75%, 25+100%, 50+75%, 50+100% e 75+100%); 3 = average values of the combination of three disks (0+25+50%, 0+25+75%, 0+25+100%, 0+50+75%, 0+50+100%, 0+75+100%, 25+50+75%, 25+50+100%, 25+75+100 e 50+75+100%); 4 = average values of the combination of four disks (0+25+50+75%, 0+25+50+100%, 0+25+75+100%, 0+50+75+100%, 25+50+75+100%). ns = not significant in tolerance level set equal to 15% of the estimated values with the models and determined from the average of five discs.

Table 4: Pearson correlation coefficients between the average value of higher calorific value of 5 disks and disk combinations along the bole to estimate the calorific value of forest species *Eucalyptus grandis* and *Acacia mearnsii*.

GCV average 5 disks	Pearson correlation coefficient							
	1 disk		2 disks		3 disks		4 disks	
E. grandis 4524 kcal kg^{-1}	0%	0.912	0+25%	0.887	0+25+50%	0.902	0+25+50+75%	0.998*
			0+50%	0.469	0+25+75%	0.292		
	25%	0.621	0+75%	-0.277	0+25+100%	0.966	0+25+50+100%	0.942
			0+100%	0.969	0+50+75%	0.871		
	50%	0.911	25+50%	0.829	0+50+100%	0.988*	0+25+75+100%	0.774
			25+75%	-0.519	0+75+100%	0.190		
	75%	-0.687	25+100%	0.886	25+50+75%	0.977	0+50+75+100%	0.928
			50+75%	0.844	25+50+100%	0.900		
	100%	0.993*	50+100%	0.963	25+75+100%	0.636	25+50+75+100%	0.995
			75+100%	-0.135	50+75+100%	0.933		
A mearnsii 4423 kcal kg^{-1}	0%	0.889	0+25%	0.999*	0+25+50%	0.998*	0+25+50+75%	0.999*
			0+50%	0.932	0+25+75%	0.998*		
	25%	0.847	0+75%	0.973	0+25+100%	0.999*	0+25+50+100%	0.998*
			0+100%	0.892	0+50+75%	0.974		

	50%	0.976	25+50%	0.975	0+50+100%	0.933	0+25+75+100%	0.997*
			25+75%	0.911	0+75+100%	0.974		
	75%	0.980	25+100%	0.839	25+50+75%	0.976	0+50+75+100%	0.974
			50+75%	0.998*	25+50+100%	0.973		
	100%	-0.663	50+100%	0.978	25+75+100%	0.907	25+50+75+100%	0.975
			75+100%	0.975	50+75+100%	0.998*		

Table 5: Pearson correlation coefficients between the average value of higher calorific value of 5 disks and disk combinations along the bole to estimate the calorific value of forest species *Mimosa scabrella* and *Ateleia glazioveana*.

GCV average 5 discs	Pearson correlation coefficient							
	1 disck		2 discks		3 discks		4 discks	
M. scabrella 4523 kcal kg^{-1}	0%	0.212	0+25%	0.767	0+25+50%	0.888	0+25+50+75%	0.995
			0+50%	0.613	0+25+75%	0.984		
	25%	0.999*	0+75%	0.962	0+25+100%	0.962	0+25+50+100%	0.983
			0+100%	0.901	0+50+75%	0.989		
	50%	0.944	25+50%	0.994*	0+50+100%	0.962	0+25+75+100%	0.998
			25+75%	0.976	0+75+100%	0.998*		
	75%	0.937	25+100%	0.989	25+50+75%	0.972	0+50+75+100%	0.999*
			50+75%	0.941	25+50+100%	0.984		
	100%	0.968	50+100%	0.962	25+75+100%	0.974	25+50+75+100%	0.971
			75+100%	0.952	50+75+100%	0.951		
A. glazioveana 4628 kcal kg^{-1}	0%	-0.491	0+25%	-0.662	0+25+50%	0.817	0+25+50+75%	0.843
			0+50%	0.429	0+25+75%	-0.088		
	25%	-0.201	0+75%	0.309	0+25+100%	-0.998	0+25+50+100%	-0.134
			0+100%	-0.627	0+50+75%	0.881		
	50%	0.723	25+50%	0.963	0+50+100%	0.061	0+25+75+100%	-0.602
			25+75%	0.083	0+75+100%	-0.909		
	75%	0.425	25+100%	-0.808	25+50+75%	0.732	0+50+75+100%	0.422
			50+75%	0.999*	25+50+100%	0.471		
	100%	-0.721	50+100%	0.274	25+75+100%	-0.266	25+50+75+100%	0.744
			75+100%	-0.452	50+75+100%	0.801		

Although there was no significant variation along the boles in this study, an increase in sample number tends lead towards the average estimates of GCV, which can be seen in Figure 1. Greater sample composition tends to nullify the effects of observed extremes and outliers regarding the influence of wood properties associated with GCV. The method used (Stepwise) proved to be efficient in estimating the predictive coefficients of the model. As mentioned by Draper and Smith (1980), the Stepwise Multiple Regression is one of the most recommended methods for a strict selection explanatory variables in the establishment of a model.

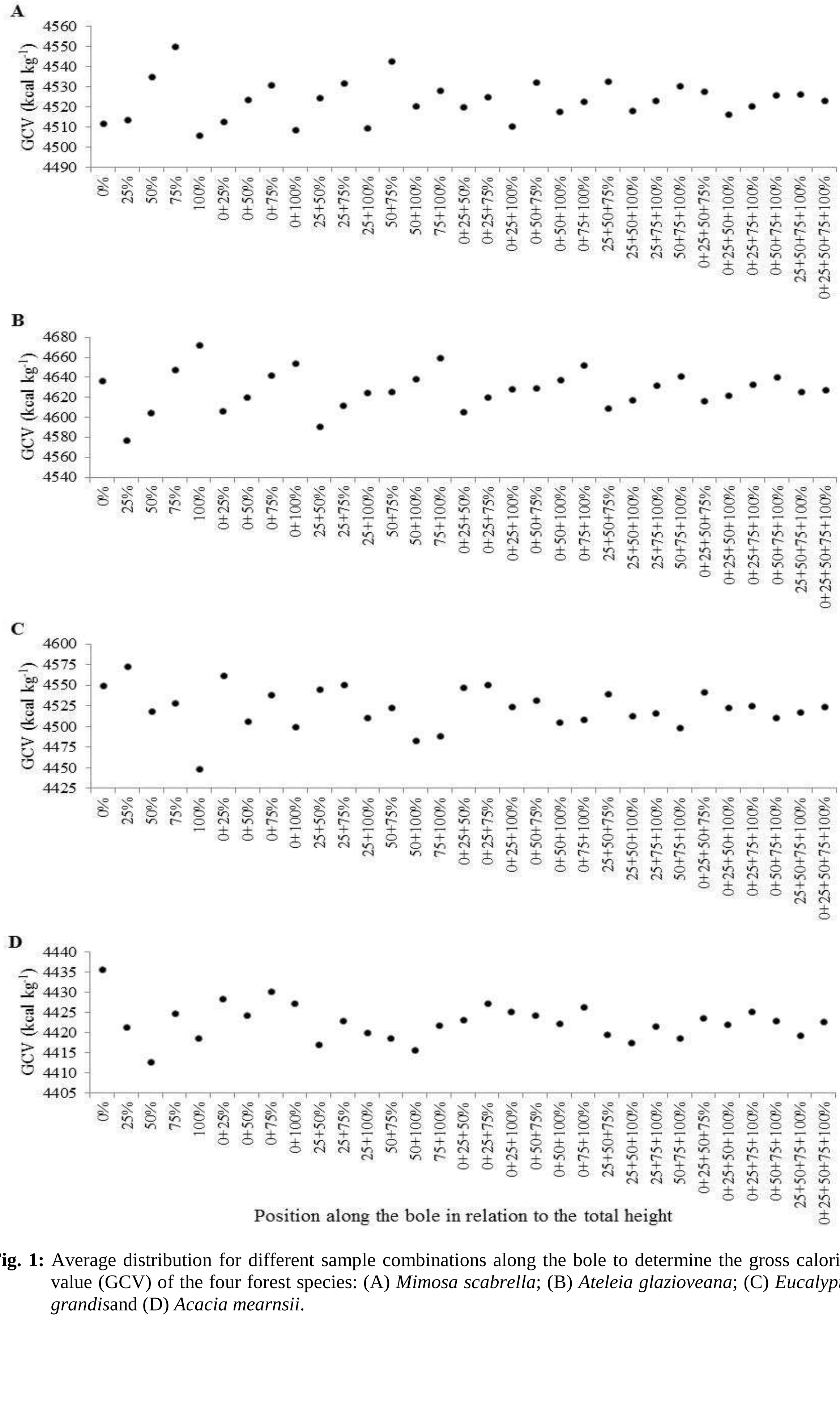

Fig. 1: Average distribution for different sample combinations along the bole to determine the gross calorific value (GCV) of the four forest species: (A) *Mimosa scabrella*; (B) *Ateleia glazioveana*; (C) *Eucalyptus grandis*and (D) *Acacia mearnsii*.

In order to achieve more accurate values, at least, two disks must be sampled in order to calculate a more accurate sampling estimate; for *Acacia mearnsii, Mimosa scabrella* and *Ateleia glazioveana,* the most ideal positions are located at 0% + 25%, 25+ 50% and 50% + 75%, respectively. For *Eucalyptus grandis* three disks at positions 50% + 100% + 0% should be used in relation the total height. In these positions, one can obtain an accurate value for the composite mean of 5 disks.

Conclusion:

Mean values for GCV for the species *Acacia mearnsii*, *Mimosa scabrella*, *Eucalyptus grandis* and *Ateleia glazioveana* are 4.423; 4.511; 4.523 and 4.628 kcal kg^{-1}, respectively.

For species *Mimosa scabrella*, *Acacia mearnsii* and *Eucalyptus grandis*, it is possible to estimate the GCV from one disk in the positions of 25%, 75% and 100%, and *Ateleia glazioveana* with two discs at the position 50 + 75%.

For a composite sample, the idea groupings of samples along the bole for *Acacia mearnsii*, *Mimosa scabrella* and *Ateleia glazioveana*, where two discs should be sampled, are the positions 0% and 25%, 25 and 50%, and/or 50 + 75%. For *Eucalyptus grandis*, the most ideal grouping of three disks at positions 0% and 50% and 100%.

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