



UNIVERSITÀ POLITECNICA DELLE MARCHE
Repository ISTITUZIONALE

Application of ISO standards on sampling and effects on the quality assessment of solid biofuel employed in a real power plant

This is the peer reviewed version of the following article:

Original

Application of ISO standards on sampling and effects on the quality assessment of solid biofuel employed in a real power plant / Toscano, G.; Leoni, E.; Feliciangeli, G.; Duca, D.; Mancini, M.. - In: FUEL. - ISSN 0016-2361. - ELETTRONICO. - 278:(2020). [10.1016/j.fuel.2020.118142]

Availability:

This version is available at: 11566/283352 since: 2024-05-16T14:49:22Z

Publisher:

Published

DOI:10.1016/j.fuel.2020.118142

Terms of use:

The terms and conditions for the reuse of this version of the manuscript are specified in the publishing policy. The use of copyrighted works requires the consent of the rights' holder (author or publisher). Works made available under a Creative Commons license or a Publisher's custom-made license can be used according to the terms and conditions contained therein. See editor's website for further information and terms and conditions.

This item was downloaded from IRIS Università Politecnica delle Marche (<https://iris.univpm.it>). When citing, please refer to the published version.

(Article begins on next page)

The published version in FUEL (Volume 278, 15 October 2020, 118142) is available at <https://doi.org/10.1016/j.fuel.2020.118142>

Application of ISO Standards on sampling and effects on the quality assessment of solid biofuel employed in a real power plant

Toscano G., Leoni E., Feliciangeli G., Duca D., Mancini M.*

Department of Agricultural, Food and Environmental Sciences, Marche Polytechnic University, Via Brecce Bianche, 60131 Ancona, Italy

*Corresponding author: Mancini M. – m.mancini@pm.univpm.it

Abstract

Bioenergy is one of the main contributors in the renewable energy markets but the quality of solid biomass is often a concern. This study provides an application of the ISO 18135 on biomass sampling, evaluating how sampling procedure affects the analytical results and suggesting possible solutions to reach an acceptable level of precision for quality assessment.

A sampling plan compliant with the aforementioned standard was carried out in a 21 MW biomass power plant located in Northern Italy in August and February, collecting samples of different typology from heaps stored in the plant and from trucks coming from suppliers. Moisture content analysis was performed for all the samples, and for those from heaps ash and nitrogen contents were also investigated.

Considering moisture content, to achieve a good precision, a significantly different number of increments are needed in August and February. Regarding ash content, it would be impossible to obtain the precision suggested by the standard, because it would lead to a number of increments not in line with practical operations. Nitrogen content is not a concern in this case, because of the measured low values. In general terms, precision values suggested by ISO 18135 require a high effort by operator and resulted unsuitable in terms of practical application for moisture and ash contents. This is probably due to the different solid biomass considered during the standard development.

For this reason, it is important to assess the quality of the different solid biofuels in order to tune the sampling procedure accordingly.

Keywords: woodchip evaluation, moisture content, ash content, nitrogen content, precision, sampling error

Abbreviations

- d_{95} : nominal top size biofuel, in mm. It is the aperture size of the sieve through which at least 95 % by mass of the material passes
- n_{\min} : minimum number of increments per (sub-) lot
- N_{SL} : number of sub-lots

- P_L : overall precision for the sampling, sample preparation and testing for the whole biofuel lot at 95 % confidence level
- V_I : primary increment variance
- V_{PT} : preparation and testing variance
- SMB: San Marco Bioenergie. It refers to the biomass power plant, monitored in this study
- CW: conifer woodchip
- BW: broadleaf woodchip
- MW: mixed woodchip
- CCWO: coarsely chopped wood from orchard (this last type of wood comes from orchard roots cleaned of the soil and subjected to a first coarse grinding before being transported to the plant- $d_{95} > 100$ mm)
- M: moisture content
- A: ash content
- N: nitrogen content
- Aug: August
- Feb: February

1. Introduction

The support schemes for bioenergy have been very successful in scaling up renewable energy deployment in the European Union and bioenergy has still remained high on the EU policy agenda to improve sustainability. Bioenergy is one of the main contributors in the renewable energy markets and has a large contribution potential for a low carbon economy [1].

In Italy, most of the electric energy production generated from power plants based on solid biofuel combustion technology is concentrated on few dozens of plants. In 2003, 27 power plants higher than 5 MWe were recorded, amounting to 257.2 MWe [2]. The same authors assessed the average biomass consumption as 10.2kt/MWe per year referred to power, and 1.44t/MWhe referred to energy production, in the operative time of 7100 h/year. The related solid biofuel use is estimated around 2.6 million tons, with over 75% being woodchip. The Association of Energy from Solid Biomass (EBS) – gathering 18 power plants greater than 5 MWe – has reached 320 MWe in 2017. Furthermore, even considering medium-low power systems, 403 plants with a total amount of 4193 GWh/year of energy production were reported [3]. Indices of solid biomass consumption to electric energy production entailed a consumption ranging from 6.0 to 7.4 million tons of biomass, mostly related to forestry (woodchip) and to agricultural operations (grinded wood from orchard).

Biomass power plants higher than 5 MWe employ between 50 and 250 kt of biomass each year. Biofuel quality control is one of the most important requirements not only for power plants, but also for other stakeholders. In general terms, the whole supply chain of bioenergy (fuel supply, combustion system, solid and gaseous emissions) is influenced by the type of solid biofuel, its physical characteristics (e.g. particle size, bulk density, moisture content (M), gross calorific value) and its chemical composition [4]. The need of evaluating solid biofuel properties is due to economic and technical-environmental reasons.

M represents a fundamental parameter. It causes the net heating value reduction due to the amount of energy absorbed during the vaporization process, thus reducing energy production [5]. The microorganism activity is also stimulated with consequent i) loss of organic substance, ii) uncontrolled combustion ignition [6]. In addition, M decreases the adiabatic temperature of

combustion, producing negative effects in terms of emissions into the atmosphere [7][8]. Considering the significant economic impact, the different power plant managers establish the biomass price based on M. Ash content (A) involves economic effects too. Indirectly, it contributes to dust emission and operational problems such as fouling, slagging and corrosion [7][8][9], on the basis of the ash chemical composition and in particular of specific chemical elements such as chloride (Cl) and sulfur (S) [6]. These elements, in addition to nitrogen content (N), cause polluting emission, as nitrogen oxides, sulfur dioxide and hydrogen chloride [9]. N concentration is variable in wooden tissue, as in bark where it is particularly present [10].

As before mentioned, the knowledge of biofuel properties is fundamental for power plants in terms of economic and environmental aspects. Some power plant managers apply biofuel quality assurance systems by assessing biomass physical and chemical features using laboratory analytical methods. The value and the effectiveness of laboratory results depend on a fundamental operative step: biomass sampling. It consists in the extraction of an amount of material from a 'lot' (or 'sampling target'), obtaining a small portion of material (sample), which can be considered to be representative of the lot [11][12]. The inherent heterogeneity of solid biofuel makes this operation complicated [13] and far from trivial, so it requires a solid understanding of both this phenomenon and how it can be counteracted in the sampling process [14].

To ensure the sample representativeness, correct sampling procedures should be followed [15]. The correct procedure for solid biomass sector is defined by technical standard: ISO 18135 – Solid biofuel – Sampling. This standard was implemented by European states and in Italy was published as UNI EN ISO 18135. Its rigorous application allows the sample preparation in several conditions for performing the following laboratory analysis. For example, the sampling can be performed where the raw materials grow, in the production plant, during deliveries (e.g. lorry loads) or from pile stocks. It includes both manual and mechanical methods and is applicable for many solid biofuel typologies [16].

Biomass sampling importance was valued by PHYDADES project [17], where it was highlighted that the deviation of analytical results from the "true" value depended by the sampling step for 80%, by the sample preparation for 15% and by the analyses only for 5%. Sampling error could increase in case of high variability of feedstock formed by heterogeneous species (conifers and broadleaves) [18].

Thanks to the multiannual experience of authors [19][20] – based on the activities carried out in Biomass Laboratory of the Polytechnic University of Marche – it can be reported poor attention of the operators about different biomass sampling aspects in Italy. In addition to the economic issues, due to the high costs of laboratory analyses and biomass sampling operations, it is recognized a poor consciousness about standard application limits with the solid biofuel employed in Italy. In fact, woodchip quality was previously assessed and resulted heterogeneous and out of quality requirements provided by European and international standards (EN ISO 17225-4 – Graded wood chips) [19]. Indeed, the main Italian power plants employed woodchip coming from forestry residues, tree crashing recovery, but also from agricultural residues, which could influence fuel quality [21] [22].

In conclusion, it was considered useful to investigate the ISO 18135 application on solid biofuel acquired by a specific biomass power plant of 21 MWe. The aims of this study are: i) to show the practical application of ISO 18135 standard in a real production context; ii) to demonstrate how the sampling procedure affects the analytical results of solid biofuel properties and iii) to suggest the suitable sampling method to define an acceptable precision level in biofuel quality assessment. To this end different scenarios have been modelled considering different operative settings of sampling.

2. Materials and methods

2.1 Sampling

Sampling procedure was applied in a 21 MWe biomass power plant, San Marco Bioenergie (SMB) located in north Italy, fed with woodchips and orchard residues within 70 km distance range from plant. The study was carried out in two moments of the year, in detail 28th August and 28th February. This time plan has the aim to consider the biomass quality in two different seasons.

SMB receives biomass from different suppliers, transported by trucks that unload the solid biofuel in a specific point in the yard of the power plant. The quantities of biofuel coming from the various trucks were separated according to their typology and form heaps of more than 100 tons.

The sampling plan was carried out using four different types of woody biomass, considered as representative of the material delivered to SMB from all suppliers: conifer woodchip (CW), broadleaf woodchip (BW), mixed woodchip (MW) and chopped wood from orchard (CCWO).

According to ISO 18135, sampling plan consists on establishing suitable sub-lots number (N_{SL}) – portion of a lot for which a test result is required – and number of increments per sub-lots (n_{min}) – portion of the biofuel extracted in a single application of the sampling device – to obtain combined sample for each sub-lot. Both numbers are associated to specific equation [16].

Sampling method is developed with N_{SL} and n_{min} parameters, from which precision (P_L) of entire analytical test on specific solid biofuel is dependent. P_L is defined by the standard as the closeness of agreement between the results by applying the experimental procedure several times under prescribed conditions. P_L values are defined by the standard for different biofuels and for different N_{SL} and n_{min} . The standard provides also an equation to calculate it [16]

According to ISO 18135, preparation and testing variance (V_{PT}) is the measure of variability associated to the overall sampling procedure. It was calculated collecting 20 subsamples composed each of 10 increments taken all around the heap in a sealed plastic bag of around 50 liters. Subsequently, each subsample was adequately mixed and divided in two smaller quantities through the quartering procedure and recorded as two samples. In fact, V_{PT} value is based on sampling and subsequent analyses error and it can be determined directly on the solid biofuel by the specific procedure. The difference between the two results has been calculated for each pair and V_{PT} can be determined with a specific equation [16].

Primary increment variance (V_I) is the measure of variability associated to sample characteristics and depends on the type and particle size distribution of the solid biofuel, on the absolute value of the parameter to be determined, on the quantity of increment taken and on the pre-treatment and mixing degree. It was calculated, according to specific equation [16], collecting 30 samples composed by each increment from all around the heap and conserved in a sealed plastic bag of around 5 liters. As reported in the standard, V_I and V_{PT} are needed to make an estimate of the precision of the experimental results.

As recommended by the standard, each increment was collected with a scoop of about 2.25 liter assuming different nominal top sizes (d_{95}): i) for CW, BW and MW, a d_{95} of about 45 mm, ii) for CCWO, a d_{95} of 100 mm. A total of 140 samples was collected for calculating V_{PT} and V_I .

In addition, the duration of sampling procedure was monitored in order to establish the minimum time required by the operator. It was calculated considering the operative variables that could slow

the sampling procedure. They can be divided into different groups: i) heap or truck characteristics (size and height, distance from storage area and particle size), ii) weather conditions, iii) operator skills and experience and iv) additional activities (labelling, biomass displacement).

2.2 Lab analysis

The quality of the collected samples was established by measuring moisture content (M), ash content (A) and nitrogen content (N), for the importance represented by these parameters and their influence on economic, environmental, and energy aspects. In fact, they represent the most important characteristics of solid biofuel, because associated to its fundamental fractions: water portion (M), organic part (N) and inorganic one (A).

All analyses were carried out according to current ISO standards about solid biofuel characterization and have been summarized in Table 1.

Table 1. Technical procedure and methodology description of the analysis carried out for each parameter.

| Analysis | Instrument | Technical standard | Basic methodology |
|--------------------|--------------------------------------------------------------------|--------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Sample preparation | Cutting mill RETSCH SM 2000 | ISO 14780:2017 | The sample is stabilized in an oven at 40 °C for about 24 h, then milled in order to obtain a particle size distribution < 1 mm. |
| Moisture content | Ventilated stove “MPM Instruments” type M 250-VF, Electronic Scale | ISO 18134-2:2015 | A sample of about 300 g is weighed and set in an oven (105 °C for 24 h), then weighed again. The percentage of evaporated water is the result. |
| Ash Content | Ash analyzer TGA 701 LECO | ISO 18122:2015 | About 1 g of milled material is weighed and brought to incineration through three steps – 105, 250 and 550 °C – in an oxidizing atmosphere until it reaches a constant weight. The inorganic fraction of the starting material is the remaining mass after the process. |
| N content | N analyzer FP-528 LECO | ISO 16948:2015 | By-products of combustion of about 0,10 g of milled material pass through a furnace filter and a thermoelectric cooler for subsequent collection in a ballast apparatus and then measured by the thermal conductivity cell for nitrogen. |

The analysis of M was carried out on MW and CCWO coming from unloaded trucks of specific suppliers, while M, A and N was carried out on CW and BW heaps.

2.3 Statistical analysis

Descriptive statistics have been carried out on all the considered parameters. Two-way analysis of variance (ANOVA) has been performed to investigate the significant differences ($p < 0.05$) between:

- August/February and CW/BW on heaps;
- August/February and MW/CCWO on truck;

All the data analysis was computed in R software (Ver.3.1.2, R development core team) equipped with the following package: car, lsmeans and multcompView [23], [24], [25].

2.4 Scenarios definition

P_L is considered as the random errors of the scheme and the lower the value, more precise the sampling performance is. Different operative settings of sampling were elaborated by using N_{SL} and n_{min} parameters combinations to obtain specific P_L value. For each parameter a scenario has been proposed as an example. The aim was establishing a P_L value acceptable from an analytical point of view and the related N_{SL} - n_{min} combination, feasible in terms of technical and economic aspects.

3. Results

3.1. Calculation of V_I and V_{PT} for each quality parameter

Table 2 shows the results of calculation of V_{PT} and V_I based on M for MW and CCWO samples on truck.

Table 2. V_{PT} and V_I results for moisture content on truck.

| Empty Cell | Empty Cell | MW | CCWO |
|------------|-----------------|-------|--------|
| V_{PT} | August | 1.205 | 11.177 |
| Empty Cell | February | 5.884 | 29.492 |
| V_I | August | 2.538 | 11.758 |
| Empty Cell | February | 8.181 | 16.669 |

M value is higher in CCWO than in MW samples, both in August and February. In addition, the difference between the two months is more evident for CCWO samples, in particular for V_{PT} value (29.492), probably because of the different particle size of the material. In fact, the loss of moisture is more difficult due to coarse particle size of CCWO samples, also considering the lower temperatures and the higher rainfalls of February.

Table 3 reports the results of calculation of V_{PT} and V_I based on M, A and N for CW and BW samples on heaps.

Table 3. V_{PT} and V_I results for moisture, ash and nitrogen contents on heaps.

| Empty Cell | Empty Cell | Moisture | | Ash | | Nitrogen | |
|------------|-----------------|----------|---------|-------|-------|----------|-------|
| Empty Cell | Empty Cell | CW | BW | CW | BW | CW | BW |
| V_{PT} | August | 1.490 | 0.477 | 0.793 | 0.592 | 0.001 | 0.004 |
| Empty Cell | February | 1.840 | 1.154 | 0.902 | 0.485 | 0.001 | 0.002 |
| V_I | August | 54.706 | 16.138 | 2.619 | 1.540 | 0.005 | 0.001 |
| Empty Cell | February | 41.302 | 102.133 | 8.216 | 0.892 | 0.009 | 0.007 |

Considering M, V_{PT} value is higher in CW than in BW samples, both in August and February. Instead, V_I value for BW (102.133) shows evident difference compared to CW in February (41.302), but it is lower (16.1385) compared to V_I value of CW in August (54.706). No specific V_{PT} and V_I values are reported by ISO standard for conifer and broadleaf woodchip, so the aforementioned difference is not taken into account.

Considering A, it is noteworthy that V_I values are higher in CW than in BW samples (especially in February). The same trend can be observed for V_{PT} values.

Considering N, the differences between CW and BW are not so evident. V_I values are higher for BW samples, the contrary for V_{PT} . To consider that N variability is not considered by the standard for woodchip.

3.2 Descriptive statistics and analysis of variance

Table 4 reports statistical results of M, A and N parameters, related to biomass typology and sampling months, with related analysis of variance carried out using Tukey test ($p < 0.05$).

Table 4. Results of descriptive statistics for moisture, ash and nitrogen contents (August = Aug, February = Feb, Standard deviation = STD, Variance = VAR, Minimum = MIN, Maximum = MAX). Mean values with the same letters are considered not significantly different with $p < 0.05$.

| Empty Cell | Moisture | | | | Ash | | | | Nitrogen | | | |
|--------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
| | <i>CW</i> <i>Aug</i> | <i>BW</i> <i>Aug</i> | <i>CW</i> <i>Feb</i> | <i>BW</i> <i>Feb</i> | <i>CW</i> <i>Aug</i> | <i>BW</i> <i>Aug</i> | <i>CW</i> <i>Feb</i> | <i>BW</i> <i>Feb</i> | <i>CW</i> <i>Aug</i> | <i>BW</i> <i>Aug</i> | <i>CW</i> <i>Feb</i> | <i>BW</i> <i>Feb</i> |
| MEAN | 28.7 ^a | 28.2 ^a | 43.8 ^b | 43.6 ^b | 3.46 ^a | 5.91 ^c | 4.53 ^b | 3.87 ^{ab} | 0.29 ^a | 0.44 ^c | 0.36 ^b | 0.45 ^c |
| STD | 2.9 | 2.3 | 3.7 | 3.1 | 0.89 | 1.42 | 1.35 | 0.85 | 0.04 | 0.06 | 0.05 | 0.06 |
| VAR | 8.2 | 5.5 | 13.5 | 9.6 | 0.79 | 2.01 | 1.82 | 0.73 | <0.01 | <0.01 | <0.01 | <0.01 |
| MIN | 23.6 | 24.1 | 37.8 | 35.3 | 1.79 | 3.26 | 2.31 | 2.69 | 0.20 | 0.35 | 0.24 | 0.32 |
| MAX | 33.4 | 32.8 | 54.8 | 52.7 | 6.10 | 9.10 | 8.71 | 7.48 | 0.36 | 0.60 | 0.44 | 0.60 |
| RANGE | 9.8 | 8.7 | 16.9 | 17.4 | 4.31 | 5.84 | 6.40 | 4.80 | 0.16 | 0.25 | 0.20 | 0.28 |

Tukey test showed significant differences in M between February and August both on CW and BW obviously because of the climate conditions. CW samples show the highest variance in February (13.5). Hygroscopic feature of wood is responsible of the great variation of M, also according to other authors [6].

Regarding A, CW and BW in August result significantly different. CW samples give the lowest value in August, while BW samples in the same month, the highest. The higher presence of leaves or green parts in BW samples in August probably increases A. For both CW and BW, in general, high values of A could be related to the presence of bark which remains included in the chipped woody material [6].

Regarding N, Tukey test showed significant differences between CW and BW samples, but also between February and August only for CW samples. In detail, CW samples in August have lower N than in February and BW samples have the highest N values. According to other studies [26], [27], N is influenced by edaphic characteristics during growth and litter decomposition velocity, so C/N

rate during conifer decomposition is higher than broadleaf [28]. As last, N values never exceed 0.5% for each biomass and season.

Table 5 reports statistical results of M parameter for MW and CCWO, related to sampling months, with related analysis of variance carried out using Tukey test ($p < 0.05$).

Table 5. Results of descriptive statistics for moisture for MW and CCWO samples (August = Aug, February = Feb, Standard deviation = STD, Variance = VAR, Minimum = MIN, Maximum = MAX). Mean values with the same letters are considered not significantly different with $p < 0.05$.

| Empty Cell | Moisture | | | |
|--------------|-------------------|-------------------|-------------------|-------------------|
| Empty Cell | <i>MW Aug</i> | <i>CCWO Aug</i> | <i>MW Feb</i> | <i>CCWO Feb</i> |
| MEAN | 28.2 ^a | 29.4 ^a | 41.8 ^b | 44.4 ^c |
| STD | 1.5 | 3.9 | 2.9 | 6.0 |
| VAR | 2.2 | 15.6 | 8.5 | 36.4 |
| MIN | 22.8 | 21.5 | 33.9 | 33.1 |
| MAX | 32.6 | 39.8 | 48.5 | 61.6 |
| RANGE | 9.8 | 18.3 | 14.6 | 28.5 |

Tukey test showed significant differences in M between February and August, both on MW and CCWO. In addition, MW and CCWO are significantly different also in February. This is probably related to the fact that during this month CCWO better retains moisture because of the higher particle size of the material.

4. Discussion

For the woody biofuel employed in Italian power plants, the evaluation of P_L requires the correct application of ISO 18135, so the calculation of V_I and V_{PT} . In fact, present results and field experience showed that the application of V_I and V_{PT} values suggested by the standard of sampling (annex D, Table D.5) can lead to substantial errors, making laboratory analyzes not meaningful and somehow misleading because the P_L proposed by sampling standard can overestimate the real one. This is particularly true when the biofuel deriving from forestry, pruning and agricultural residues presents a certain degree of heterogeneity and particle size different from the one reported in the standard.

In this study V_I and V_{PT} values calculated for M and A are considerably higher than those reported on the standard of sampling for the same biofuel typology. As a consequence, to obtain P_L values similar to those suggested by the standard, the sampling plan should require a higher n_{min} and N_{SL} , with consequent operative costs.

For example, considering M as the main variable parameter between CW and BW samples collected from heap, performing a sampling with $N_{SL} = 3$, in order to obtain $P_L = 2.0\%$ the sampling should consist of over 50 increments in the winter season, but less than 8 increments in the summer one.

This can be easily verified by looking at Figure 1 where the results, obtained applying the equations connecting P_L , N_{SL} and n_{min} , are reported.

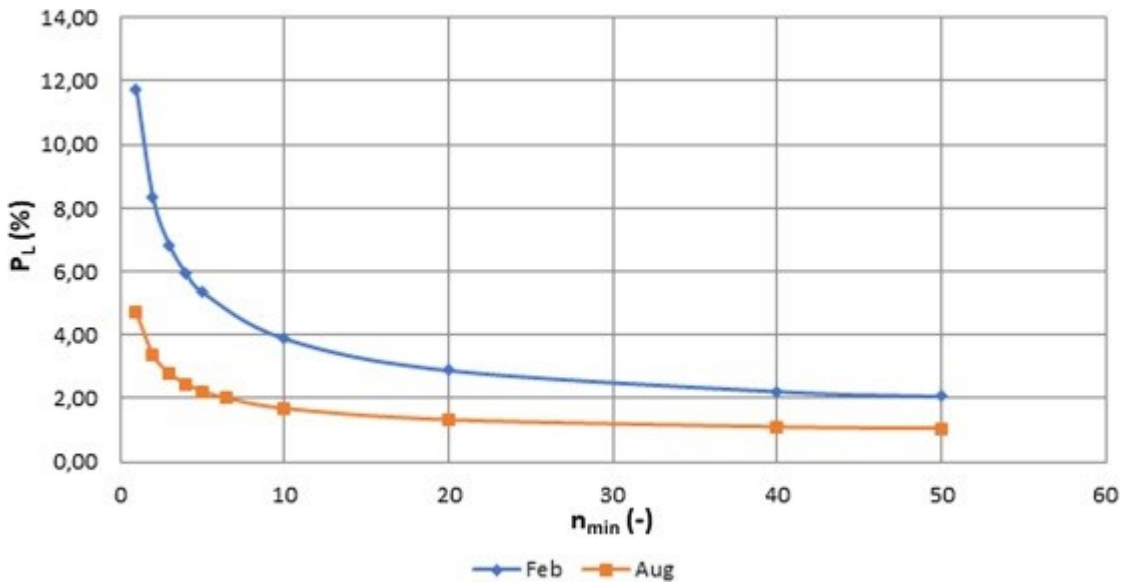


Figure 1: representation of P_L as a function of different values of n_{min} and $N_{SL} = 3$ in the case of the sampling from heap of BW in August and February for M. The black lines mark the minimum n_{min} to obtain $P_L=2$.

Using the same methodology of the previous example, to obtain a $P_L = 1\%$ - indication suggested by the standard of sampling – the sampling would require a consistent effort, especially in the case of CW. In fact, this solution would entail a sampling of $N_{SL} = 8-9$ and n_{min} between 50 and 100 (Figure 2).

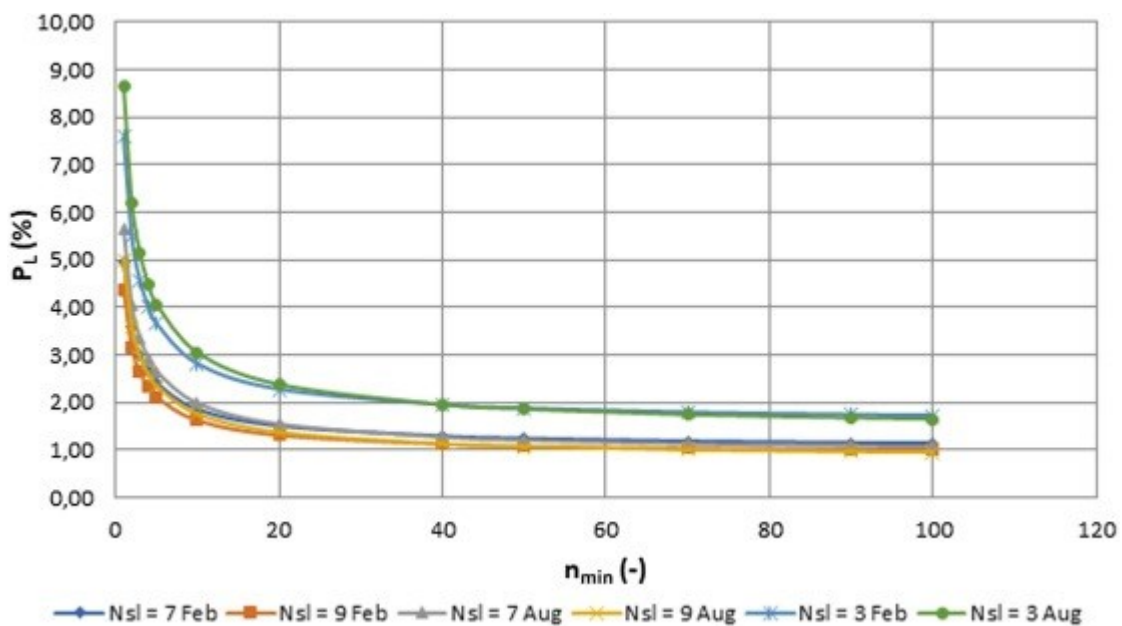


Figure 2: Graphic representation of P_L as a function of n_{min} and different N_{SL} from the sampling from heap of CW in August and February for M.

According to the sampling operational time, calculated based on criteria described in chapter 2.1, to achieve this result a team of two operators would employ at least 7-8 working hours, with important consequences on analytical costs that become unacceptable for the power plants, making the sampling and analysis nearly impractical. Consequently, both in August and February sampling procedure turns to be feasible with $N_{SL}=3$ and $n_{min}=20$, employing about 1-2 working hours and obtaining an acceptable P_L around 2.3÷2.4%.

The data emerging from the sampling on trucks are partly consistent with those already described for heaps. As shown in table 2 with respect to V_I and V_{PT} values for M, CCWO is more variable than MW samples, both in February and August. In the case of MW, by applying the sampling procedure with the same values of N_{SL} and n_{min} , the resulting P_L value is around twice higher in February compared to August. Instead, in case of CCWO, P_L shows a reduced difference between the two months, usually more than 1.5 times. For example, considering $N_{SL}=12$ and $n_{min}=25$, P_L is equal to 2% in August and 3.2% in February.

The strong differences in V_I and V_{PT} between MW and CCWO are most likely attributed both to the typology of wood and to its particle size.

According to the results, it is impractical to apply a sampling with P_L value of 1% because of the high values of N_{SL} and n_{min} required, with the exception of BW and MW samples in August. Nevertheless, a good precision ($P_L=1.8\div 2.1\%$) can be obtained through a practical sampling procedure, i.e. $N_{SL}=4$ and $n_{min}=30$.

Also for A parameter, it is impossible to obtain P_L value of 0.1% as suggested by the standard, because it would lead to $N_{SL}>10$ and $n_{min}>100$. For example, in the case of BW, P_L values of 0.6-0.9% can be obtained with $N_{SL}=3\div 5$ and $n_{min}=20\div 30$ (Figure 3). Taking into account the ISO 17225-4 requirements for B class woodchip ($A \leq 3.0\%$) and the mean value resulting from this study of $4.4 \pm 1.1\%$, it could be acceptable to consider also a sampling with $P_L = 0.9\%$.

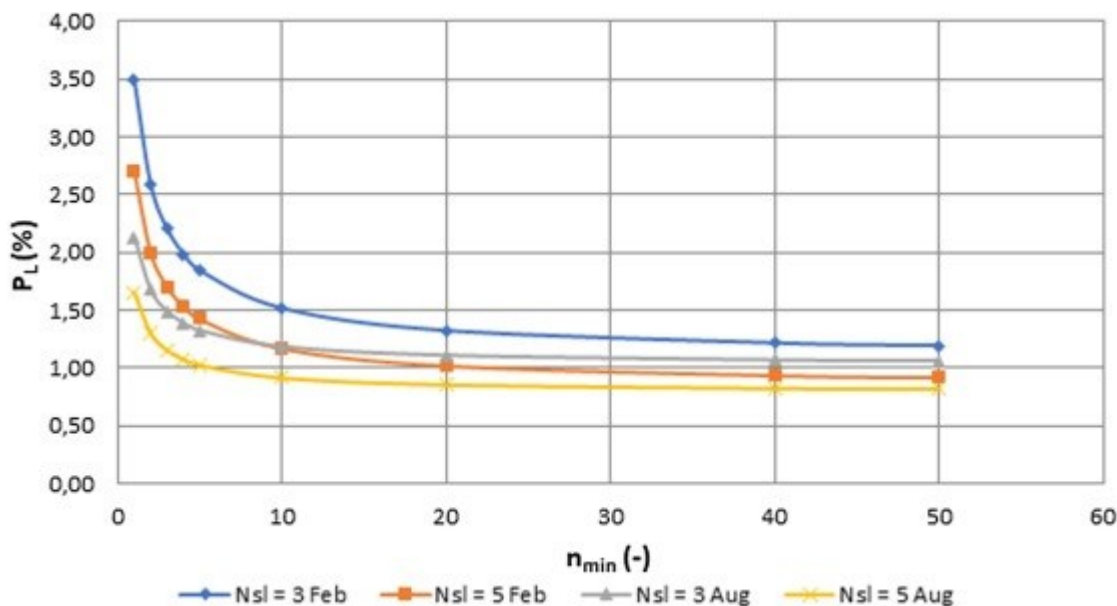


Figure 3: Graphic representation of P_L as a function of n_{min} and different N_{SL} from the sampling from heap of CW in August and February for A.

Regarding N parameter, standard does not consider P_L values for woodchip. A sampling with $N_{SL}=2$ and $n_{min}=10$ allows to obtain P_L lower than 0.1% (Figure 4). Taking into account the ISO 17225-4 requirements for B class woodchip ($N \leq 1.0\%$) and the mean value resulting from this study $N = 0.39 \pm 0.05\%$, P_L is not a concern, especially considering the high quality of SMB woodchip in terms of N value. Nevertheless, considering the high variability in their elemental composition, it would be advisable the evaluation of V_{PT} and V_I for N parameter related to specific solid biofuel (herbaceous, fruit and seed residues).

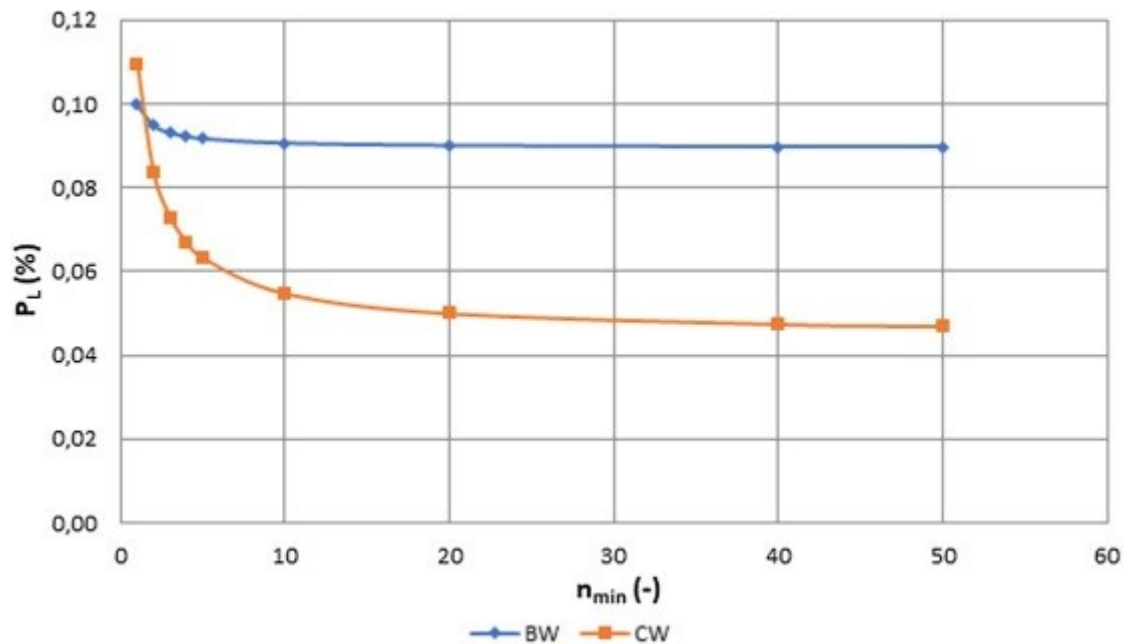


Figure 4: Graphic representation of P_L as a function of n_{min} and $N_{SL}=2$ from the sampling from heaps of BW and CW in August for N.

5. Conclusions

The present study investigated the practical application of sampling procedure in a 21 MW power plant, showing the critical issues and assessing the woodchip quality in two different months.

According to the results of V_{PT} and V_I calculation, P_L values suggested by ISO 18135 require a high number of N_{SL} and n_{min} , unsuitable in terms of practical application for moisture and ash contents. Furthermore, the V_{PT} and V_I values reported in ISO 18135, if applied to the present case study, returned P_L values significantly higher than the one defined by the standard. It demonstrates how the standard underestimates the sampling error. It also indicates that it would be useful to deepen the knowledge of the quality of Italian woodchip to correctly evaluate the related P_L .

The guidelines suggested by BIONORM project [29] and parameters proposed by ISO 18135 were limited to specific backgrounds related only to few geographical areas and wood species. For this reason, it is important to assess the quality of the different solid biofuels in order to set V_{PT} and V_I values in line with the operative conditions of the plant. In order to allow suitable sampling in terms of operative time and costs, P_L should be chosen to obtain acceptable values of N_{SL} and n_{min} , even increasing P_L values. In addition, the PHYDADES project, focused on defining the sampling relevance, established that results are affected for 80% by the sampling operation. These

observations, confirmed by the results of the present study, lead also to reconsider the current approach of certification schemes on solid biofuel sector, recommending more attention to the sampling procedure, also applying the ISO 18135 procedure to determine the correct values of V_I and V_{PT} , than focusing mainly on the laboratory analyses.

Nevertheless, an appropriate sampling plan, in line with ISO 18135, could involve an important commitment by the power plants in terms of time and costs. On the basis of all these considerations, it emerges also the need of rapid and less expensive measurements for assessing the heterogeneity of the solid biofuel, making the results more realistic and useful for the power plant. Future developments could move in this direction, working on the implementation of faster analytical methods such as the near-infrared spectroscopy, especially for fundamental parameters like moisture and ash content.

Acknowledgement

The authors are grateful to San Marco Bioenergie for providing the information and support needed for conducting the study.

References

- [1] Banja M, Sikkema R, Jégard M, Motola V, Dallemand JF. Biomass for energy in the EU – The support framework. *Energy Policy* 2019;131:215–28. doi:10.1016/j.enpol.2019.04.038.
- [2] Botta G, Brignoli V, Alberti M, Riva G, Scrosta V, Toscano G. *Analisi Delle Iniziative Per Elettrica Da Biomasse Agro – Industriali in Italia*. IV Convegno Nazionale Utilizzazione Termica Dei Rifiuti, Abano Terme: 2003.
- [3] GSE GSE. *Rapporto Statistico 2017-Fonti Rinnovabili*. 2017.
- [4] Obernberger I, Brunner T, Ba G. Chemical properties of solid biofuels — significance and impact. *Biomass and Bioenergy* 2006;30:973–82. doi:10.1016/j.biombioe.2006.06.011.
- [5] Korus A, Szle A. The effect of biomass moisture content on the IGCC efficiency. *Biomass and Bioenergy* 2015;80:222–8. doi:10.1016/j.biombioe.2015.05.014.
- [6] Food and Agriculture Organization of the United Nations. *Wood Fuels Handbook*. vol. 53. 2008. doi:10.1017/CBO9781107415324.004.
- [7] Van Loo S, Koppejan J. *The handbook of biomass combustion and co-firing*. London; Sterling, VA: Earthscan; 2008.
- [8] Deboni LT, Simioni FJ, Brand MA, Lopes GP. Evolution of the quality of forest biomass for energy generation in a cogeneration plant. *Renew Energy* 2019;135:1291–302. doi:10.1016/j.renene.2018.09.039.
- [9] Fournel S, Palacios JH, Morissette R, Villeneuve J, Godbout S, Heitz M, et al. Influence of biomass properties on technical and environmental performance of a multi-fuel boiler during on-farm combustion of energy crops. *Appl Energy* 2015;141:247–59. doi:10.1016/j.apenergy.2014.12.022.

- [10] Martin AR, Gezahegn S, Thomas SC. Variation in carbon and nitrogen concentration among major woody tissue types in temperate trees. *Can J For Res* 2015;45:744–57. doi:10.1139/cjfr-2015-0024.
- [11] Esbensen KH, Wagner C. Theory of sampling (TOS) versus measurement uncertainty (MU) - A call for integration. *Trends Anal Chem* 2014;57:93–106. doi:10.1016/j.trac.2014.02.007.
- [12] Theodorou D, Liapis N, Zannikos F. Estimation of measurement uncertainty arising from manual sampling of fuels. *Talanta* 2013;105:360–5. doi:10.1016/j.talanta.2012.10.058.
- [13] Pazó J a., Granada E, Saavedra Á, Patiño D, Collazo J. Heterogenic solid biofuel sampling methodology and uncertainty associated with prompt analysis. *Int J Mol Sci* 2010;11:2118–33. doi:10.3390/ijms11052118.
- [14] Wagner C, Esbensen KH. A critical review of sampling standards for solid biofuels - Missing contributions from the Theory of Sampling (TOS). *Renew Sustain Energy Rev* 2012;16:504–17. doi:10.1016/j.rser.2011.08.016.
- [15] Zhu Q. Coal sampling and analysis standards. 2014.
- [16] ISO 18135:2017 Solid Biofuel - Sampling. 2017.
- [17] Burvall J, Wiik C, Moilanen A, Alakangas E, Englisch M. Sampling and sample reduction of solid biofuels. *Www.phydades.info n.d.:*1–39.
- [18] Aminti G, Cinotti A, Lombardini C, Spinelli R, Picchi G. Industrial stress-test of a magnetic resonance moisture meter for woody biomass in southern European conditions. *Fuel Process Technol* 2018;178:189–96. doi:10.1016/j.fuproc.2018.05.032.
- [19] Toscano G, Duca D, Pedretti EF, Pizzi A, Rossini G, Mengarelli C, et al. Investigation of woodchip quality : Relationship between the most important chemical and physical parameters. *Energy* 2016;106:38–44. doi:10.1016/j.energy.2016.03.037.
- [20] Duca D, Toscano G, Pizzi A, Rossini G, Fabrizi S, Servili A, et al. Evaluation of the characteristics of vineyard pruning residues for energy applications : effect of different copper-based treatments on chemical use on com m Treatments in the vineyard er c ia l u se Analyses performed on samples. *J Agric Eng* 2016;XLVII:22–7. doi:10.4081/jae.2016.497.
- [21] Picchi G, Lombardini C, Pari L, Spinelli R. Physical and chemical characteristics of renewable fuel obtained from pruning residues. *J Clean Prod* 2018;171:457–63. doi:10.1016/j.jclepro.2017.10.025.
- [22] Anerud E, Jirjis R. Fuel quality of Norway spruce stumps – influence of harvesting technique and storage method. *Scand J For Res* 2011;26:257–66. doi:10.1080/02827581.2011.561807.
- [23] Graves S, Piepho H-P, Selzer L, Dorai-Raj S. MultcompView: Visualisations of Paired Comparisons. CRANR 2015. <https://cran.r-project.org/web/packages/multcompView/index.html>.
- [24] Lenth R. LSMeans: Least-Squares Means. CRANR 2015:20–3. <https://cran.r-project.org/web/packages/lsmmeans/index.html>.

- [25] Fox J, Weisberg S. *An R Companion to Applied Regression*, Second Edition. Sage Publications 2011. <https://CRAN.R-project.org/package=car>.
- [26] Reich PB, Walters MB, Tjoelker MG, Vanderklein D, Buschena C. Photosynthesis and respiration rates depend on leaf and root morphology and nitrogen concentration in nine boreal tree species differing in relative growth rate. *Funct Ecol* 1998;12:395–405. doi:10.1046/j.1365-2435.1998.00209.x.
- [27] Reich PB, Walters MB, Kloeppel BD, Ellsworth DS. Different photosynthesis-nitrogen relations in deciduous hardwood and evergreen coniferous tree species. *Oecologia* 1995;104:24–30. doi:10.1007/BF00365558.
- [28] Panzacchi P, Scandellari F, Tonon G. Contributo della decomposizione della lettiera alla nutrizione minerale delle colture arboree. *Italus Hortus* 2008;15 (1):37–48.
- [29] II B. Pre-normative research on solid biofuels for improved European standards. 04347 Leipzig, Germany: n.d.