Performance of a portable NIR spectrometer for the determination of moisture content of industrial wood chips fuel

Fuel

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Corresponding Author:	Gianni Picchi, Ph.D. CNR: Consiglio Nazionale delle Ricerche Sesto Fiorentino, ITALY
First Author:	Giuseppe Toscano, Professor
Order of Authors:	Giuseppe Toscano, Professor
	Elena Leoni, MSc
	Thomas Gasperini, MSc
	Gianni Picchi, Ph.D.
Abstract:	The environmental policy of the European Union is boosting the development of renewable energies. Among these, bioenergy holds the main share and is expected to further increase. Such development requires a higher degree of efficiency in the whole supply chain. This is achieved also with an enhanced fuel quality control and a better matching with the energy conversion systems. For solid biofuels, moisture content is the main quality parameters, influencing the sustainability of the whole energy system. With the aim to provide a real-time and portable tool for moisture measurement, a hand-held near infrared spectrometer was tested on a dataset of 817 woodchip samples provided by an industrial facility. A set of key performance parameters were used to compare the estimation of three alternative prediction models and the standard oven dry method. Results show a satisfactory reliability with R 2 ranging from 0.86 to 0.89 depending on the model. A single measure can be acquired in few seconds, and the potential to deploy the non-destructive analysis directly at the fuel storage (yard) and at different steps of the supply chain discloses a wide range of options to efficiently control fuel quality.
Suggested Reviewers:	Daniel Kuptz, Dr. Researcher, TFZ - Bayern (www.tfz.bayern.de) daniel.kuptz@tfz.bayern.de Researcher in a “woody biofuels” laboratory (TFZ), is very active in biomass storage and quality assessment, having tested several methods for moisture content estimation in wood chip fuels and combustion behavior of stored biofuels.
	Ana Garrido Varo, Dr. Professor, Universidad de Córdoba: Universidad de Cordoba pa1gavaa@uco.es Former president of the International Council for NIR Spectroscopy is a forefront academic in the application of NIR technology to the assesment of quality parameters on a wide range of organic products.
	Blas Mola Yudego, Dr. Professor, University of Eastern Finland - Kuopio Campus: Ita-Suomen yliopisto - Kuopion kampus blas.mola@uef.fi Professor of forest biomass production and bioenergy systems
	Johanna Routa, Dr. Researcher, Luke: Luonnonvarakeskus johanna.routa@luke.fi Lead researcher in LUKE, member of the forest biomass laboratory is specialized in biomass quality assessment and enhancement.
	Mauricio Acuna, Dr.

	Professor, University of the Sunshine Coast macuna@usc.edu.au Academic specialized in in biomass production and sensors deployed along the supply chain. Tested NIR technology for assessing the quality of timber for pulp production.

Dear editor, dear reviewers,

first of all, thanks for the time dedicated to evaluate and improve our manuscript.

Below you will find your comments listed in order of reviewer (with the numbering that appeared to us).

Each comment had been assigned to a progressive ISSUE-number and addressed by a discussion/answer reported below.

Reviewer 1

1.1. Maybe some information on how the different fuel samples (e.g. poplar wood, agricultural residues, coniferous wood, etc.) are distributed among the MC classes are available? If so, it could be of interest to compare if there is some clustering of some fuels in certain MC classes (e.g. poplar in the MC class > 60%)? Maybe the difference the authors identified for the three models in different MC classes might somewhat be related to the fuel type? If possible, please add some information to the text.

DISCUSSION TO ISSUE 1.1:

We agree with the reviewer: this aspect is highly important (at least in potential). Yet, the specific info about the type of different biofuel analysed was available just for few samples. In order to have a reliable dataset, this info should be transmitted by the wood chips producers to the power plant and from this to the biomass laboratory. At present a reliable tool for such data exchange is missing. Additionally, the description would be subjectively reported by the chipper operator, who is often working with a mix of raw materials. As a result, given the reliability and inconsistence of these information for the whole dataset, we preferred not take it into account on the few samples where it was available.

As it would be very useful to include these variables into the models, we will seek for the possibility to address them in future researches. As suggested, a brief explanation has been added in the manuscript (lines 125-128, 177-178, and 268-270, but also 391-393).

1.2. "A portable NIR spectroscope was used to estimate moisture content of biomass fuels"
"Validity, accuracy and precision of 3 prediction models were compared"
"Moisture content estimation with portable NIR is reliable, fast and non-destructive"
"Results of the prediction models differed mostly on extreme moisture content values"
"NIR spectroscopy is highly suitable to analyze fuel quality along the supply chain"

DISCUSSION TO ISSUE 1.2

Thanks for checking also this part of the submission (few reviewers pay attention to highlight or other elements besides the manuscript). We agree with the suggested changes and included them in the new highlights.

1.3. Also, the title uses the term "wood chips" while in the text, the term "woodchips" is used.

DISCUSSION TO ISSUE 1.3

This is a good point. In order to clarify which alternative was more appropriate we checked the previous publications of FUEL and found 82 occurrences for “woodchips” and 670 for “wood

chips". As a result, we used "wood chips" throughout the manuscript.

1.4. The introduction gives a good idea on why the study is important. During reading, I was wondering, if the term "portable NIR device" just relates to the portability (i.e. for handling of the sensor in the field) or if it also relates to a rather low-cost device. Many NIR devices are very expensive and maybe their costs would be too much for medium sized wood chip producers? In line 85 to 91, you also give the idea of implementing a portable device into a chipper. Thus, this device would not be "portable" anymore. Maybe, you can specify in the introduction if "portable device" refers only to handling or also to the costs (and/or robustness) of the instrument?

DISCUSSION TO ISSUE 1.4

This issue was raised also by another reviewer, highlighting how unclear we were regarding the description of the device.

For "portable" we meant a light, small sized and rugged device with internal battery. This makes it possible to operate the NIR sensor indifferently in laboratory, in the fuel yard or in the forest (among other settings). We integrated the text to make it more clear and added a couple of images (lines 156-157). As stated in the additional text, the model used is interfaced to a rugged tablet via USB connection, but a Bluetooth model is also available (lines 137-139). We also added the cost of the device. Although it is difficult to define a unique value with dealers (there is always a kind of bargaining involved) we reported the cost faced by our laboratory: 10,000 €. In our experience with power plants, this can be regarded as an average cost: cheaper solutions are available, but also much more expensive sensors can be deployed (about 60-70,000 € for magnetic resonance).

As you correctly state, once the NIR is deployed on a chipper, it is no longer "portable". Our sentence was misleading as we meant that once that the models are developed, these can be deployed in NIR sensors (portable or not) at different stages of the supply chain.

We changed accordingly the sentence (lines 92-94)

1.5. Line 65: "…wind that generally leads to MC increase."

DISCUSSION TO ISSUE 1.5

We apologize. The sentence had a typo error, making it unpleasant to read. Additionally, the "wind" factor listed after rain and snow was confusing as these are cause of MC increase, while the former reduces it. The sentence has been slightly corrected accordingly.

1.6. Line 67: Please give information what the effects of an "unstable and inefficient firing process" are, e.g. increased gaseous and particulate emissions due to cooling of the combustion chamber, etc.

DISCUSSION TO ISSUE 1.6

The sentence has been integrated as requested (lines 67-69).

1.7. Line 72: "…according to biomass quality."

DISCUSSION TO ISSUE 1.7

Thanks for identifying this typo error. The sentence had been completed.

1.8. Line 94: please add the abbreviation KPI to the text the first time you use "key performance indicators"

DISCUSSION TO ISSUE 1.8

KPI abbreviation had been added at the first appearance of the definition of "key performance indicators".

1.9. What size does the power plant (in kW heat and electricity output) have? Do you have some idea on the overall demand of wood fuels per year?

DISCUSSION TO ISSUE 1.9:

The key figures of the power plant had been added in the text (line 120).

1.10. Can you please give a rough estimate for the individual size of the wood chip samples (line 110 to 118)? And could you please give the size of the woodchip tray (length x width x height in Figure 1)? How thick was the layer of wood chips in the tray and what was the distance between measuring points? How important is the angle of the instrument above the woodchips?

DISCUSSION TO ISSUE 1.10:

Samples had an approximate fresh weight of 500 g, the figure had been added in the initial description of the samples (line 129). The size of the tray had been added in the description of the analysis procedure (line 149) as well as the depth of the wood chips layer and the reading angle.

1.11. Line 110 to 114: Can you allocate the 817 samples to the different fuel types? If so, maybe you can also see if the NIR sensor works differently for the different fuels (e.g. conifer wood compared to poplar clones)?

DISCUSSION TO ISSUE 1.11:

As commented above in the issue 1.1, the lack of specific info about the type of different biofuel analysed and the possible risk of mixed biomass in the same sample led the authors to not consider this descriptive factor. We added it as one of the goals of future researchers and developments.

1.12. Does the NIR sensor require direct contact to the woodchips? If not, what distance was used between woodchips and sensor for the measurement?

DISCUSSION TO ISSUE 1.12:

As commented in the issue 1.10 considering analysis procedure, in order to reduce light interference, the instrument approach requires direct contact to the woodchips if operative conditions being optimal. This detail had been added at lines 152-153 of the revised manuscript.

1.13. Line 130: "for" instead of "For"

DISCUSSION TO ISSUE 1.13

The typo error had been corrected as suggested.

1.14. Line 132: "by the same operator" instead of "unique operator"

DISCUSSION TO ISSUE 1.14

Corrected as suggested

1.15. Line 157: please use "Partial Least Square regression (PLS) model" when you mention PLS the first time in the text

DISCUSSION TO ISSUE 1.15

Thanks, we missed the definition of the acronym (too used to it). It had been added to the text.

1.16. Line 169: "difference in moisture content"

DISCUSSION TO ISSUE 1.16

“content” had been added as suggested.

1.17. Line 173: italics used for "and MC_ref"

DISCUSSION TO ISSUE 1.17

The italic format had been brought to normal format for the words "and MC_ref"

1.18. Can you provide somewhere in the manuscript some rough estimate on the costs of the portable NIR device? For application by the industry, low-cost and robust devices would be required. So this information might be of interest to the reader.

DISCUSSION TO ISSUE 1.18:

The unitary cost of the sensor had been added to its initial description (line 139).

1.19. Line 247 to 251: The samples with very high MC > 60% might also be dominated by poplar clones from coppice and might not derive e.g. from conifers? Thus, you might have an additional variation due to tree species? Maybe some information on the distribution of different woodchip fuels in MC classes is available? Where do the fuels with low MC derive from? Is this material stored some time before delivery to the plant? I guess this would include natural drying processes but no technical drying?

DISCUSSION TO ISSUE 1.19:

As explained by the authors in issue 1.1 and 1.11, considering the lack of information about biofuel type of each sample and its reliable origin for the whole dataset, authors have decided to not take into account this matter. Once in the yard the fuel is mixed making impossible to track the storage history of single lots. Moreover, the information about storage duration and drying prior to

delivery to the power plant is held by a plethora of providers: a direct contact with them would be necessary in order to collect reliable data. For practical reasons we focused this study on the simple MC heterogeneity of wood chips. But further studies including the type of biomass and its influence on MC estimation would be extremely valuable, even if quite difficult to establish with such a large database. Explanation has been added in the manuscript at lines 125-128 of the revised manuscript.

1.20. Line 257: I would not consider MOD1 to underestimate samples > 60% MC but that variability strongly increases in both directions (over- and underestimation, see also Figure 2). This might also relate to tree species, e.g. when poplar might overestimated at high MC but other fuels might be underestimated?

DISCUSSION TO ISSUE 1.20:

Thanks for correcting this statement. You are right, at high MC values MOD1 is basically boosting its variability. The text had been revised accordingly (lines 288-290).
Regarding the possible impact of the species composition, as stated in the previous points, this info was not available. From the model development point of view, it would be very useful and we hope to include this aspect in our future studies. However, from the practical application point of view, we aim at developing a model reliable enough to be operated without this info. This is because this data can be reliably collected just at the biomass source (say at the wood chipper), and even here it can be tricky if a mixture of species is chipped together.

1.21. Figure 4: The central line is not dashed?

DISCUSSION TO ISSUE 1.21

Correct. The error is due to a change in graphical choice not followed by caption adaptation. The caption (now Figure 5) had been corrected accordingly.

1.22. Line 326 to 329: This section on the practical applicability could be elaborated in more detail. For instance, the process chain can be optimized by measuring a much higher share of MC samples from a delivery in a shorter amount of time compared to the usual procedure using a drying cabinet and the higher sample number strongly improves the MC determination. I think that is what you mean by "avoiding sampling time and minimizing the risk of sampling errors.", but maybe describe the benefits from using a NIR sensor on-site in more detail. Also, would you consider MC determination using the NIR sensor a good alternative compared to the ISO standard method for pricing of the fuels? Moreover, depending on the costs for the sensor, it might be used by a high share of different companies but if the sensor is expensive, maybe it is only suitable for larger plants?

DISCUSSION TO ISSUE 1.22

Thanks to rise this point. We agree that the potential of the sensor is enormous. Being portable, we can perform a large number of analysis directly on the spot. While this returns a precision comparable to the oven method, it avoids the transfer of large quantities of samples. In turn, this reduces the risk of laboratory errors and of sample alteration (e.g. drying during shipment). Additionally, it provides a MC estimate with a much lower energy requirement (compared to

hundreds of samples transported and dried in the oven). We further developed these aspects in lines 364-371 of the revised manuscript.

1.23. Line 339 to 341: Maybe the fuels used to develop the models also were more homogeneous in terms of tree species?

DISCUSSION TO ISSUE 1.23:

The biomass samples used to develop the models were provided the year previous to the study by two power plants (including the one providing later the 817 samples used in the study) over a similar time frame, thus covering a similar seasonal variability. The two power plants are located in the same area and are fed with a similar mix of fuels. Considering this, and since there was no way to reliably determine the species composing the samples, we assumed that they had a similar homogeneity in MC distribution and species composition. We included this description in lines 175-181.

1.24. If you are considering future studies, you could also be interested in other external effects such as tree species, assortment, or different operators. Maybe you could improve your models further if you know the fuel type more precisely? Also, in your study, the same operator used the sensor but how repeatable would the measurement be if different people use the sensor?

DISCUSSION TO ISSUE 1.24:

Thanks, the points you rise are both relevant. Regarding the tree species/assortments, we would like to include them in the modelling stage as well as in the model validation. Yet, as previously mentioned, it is utterly difficult to compose a large dataset of samples covering a wide range of variability and having this info available in a reliable way. Generally, the facility manager trusts on the provider and the experience of the yard manager to assess the composition of the wood chips. Yet, the degree of approximation, acceptable for their purposes, would be excessive for modelling. And collecting a relevant number of samples from the wood chipper's mouth would be costly and time demanding (unless one collects a lot of samples of the same biomass).

As a result, we are still seeking for the practical solution allowing us to gather a large dataset of biomass samples with a full and reliable descriptor of species, assortments, storage history, type of chipper used, etc.

Different is the comparison of operators. This is a fairly good point. We will soon set up a study comparing the results of several operators using the same hand-held sensor on the same data pool. This had been briefly mentioned in the Conclusions (lines 389-393).

Reviewer 2

2.

2.1. A language check is recommended as well.

DISCUSSION TO ISSUE 2.1

Thanks. Indeed, the text needed a fresh-minded revision, which had been conducted thoroughly for the whole manuscript.

2.2. Several literature is cited, but a bit more specific information about NIR systems operated at practice is missing. It is suggested to provide for e.g. 2 examples some basic information about the type of material which can be measured, accuracy, performance, limitations etc. to get an idea of the current state of the development in NIR moisture measurements.

DISCUSSION TO ISSUE 2.2:

As suggested, three additional references about the optimal NIR application on natural biomass to assess moisture content had been added in the manuscript at lines 77-79 and 81-82.

2.3. Line 97: What are such factors others than the desired ones? Please elaborate this a bit more e.g. in the text before.

DISCUSSION TO ISSUE 2.3

We revised the text as suggested. The “other factors”, now called “non-controlled factors” are in this case mainly trees species and woody assortments used for the production of wood chips (lines 104-105).

2.4. Line 110-118: Make clear, which samples have been used for the measurements and built the basis for the results presented later for the different models MOD 1 - MOD 3. How large was one sample? Were these samples of mixed materials (different tree types)? Did they contain bark, leaves, needles because someone would expect that this makes an important difference for the accuracy etc. of the NIR and the model results?

DISCUSSION TO ISSUE 2.4

As stated in section 2.4 of the manuscript, the models were developed on different sets of industrial woodchip samples. These were provided by two power plants, including the one that provided (in a second moment) the 817 samples used for this study. The text had been revised in order to better clarify the origin of the different datasets used for model building and for the present study.

Unfortunately, data regarding the specific species and assortment composition of the wood chips was not available (this observation was made also by Reviewer 1). The samples used for model building and for this study were provided by power plants that receive fuel from several contractors. These may generically provide a descriptive of the feedstock. Yet, this description is always very vague and available just for a restricted number of samples. This is enough for the

purposes of the power plants, but we considered this info of a quality and consistency not sufficient for model building nor for the following validity analysis.
Including these details in future studies is among our plans, as reported in the integrated Conclusions.
On the other hand, the purpose of the research was first to develop and the to test robust models that can provide a reliable response even without information regarding the nature of the wood chips (as long as they are uncontaminated biomass). In fact, very rarely the biomass manager of the facility will have access to this info (composing species, assortments, etc.) for the incoming biomass. Thus, the practical objective was to develop a fast method that can provide a MC estimate with good approximation even without any further data regarding the fuel.

2.5. Line 117, 118, 134: Standards should be included into the list of references.

DISCUSSION TO ISSUE 2.5:
As suggested, standard have been included in the list of references and in bibliography.

2.6. Line 121: More information about the used system is required here such as the name of the producer, country of origin, model no.. Furthermore, what means portable? Is it a handheld device or do you need a transport equipment for moving? If easy handling is later an important KIP, some information about size, weight, energy supply are required here. Maybe a picture of this system could answer all these questions. Furthermore, when it comes later to the application of the calculation models: Are these models implemented into the device, can you choose between them, or do you need to transfer the raw data to a computer for subsequent calculation of the MC?

DISCUSSION TO ISSUE 2.6:
As suggested, more information about the instrument had been added at lines 134-139 and two pictures had been added as Figure 1.
All the models are run simultaneously on the table interfaced with the sensor, showing in real time the three MC estimates. A brief description had been added in lines 173-174.

2.7. Fig 1: How large is the woodchip tray and how much material (e.g. volume) is required for one measurement? How large is one measuring spot on the tray? Please provide more information to make it possible for the reader to get a decent idea about the procedure applied.

DISCUSSION TO ISSUE 2.7:
Thanks for highlighting this important missing info. It was pinpointed also by another reviewer and it was definitely too relevant to be omitted.
The size of the tray and the depth of the layer had been added in lines 149-150. Samples had a mass of about 500 g (added in the previous description of the samples, line 129). The measuring spot covers an area of about 250 mm^2 (line 154).

2.8. Line 148 and following: Are all samples (n= 817 right?) just measured once and the resulting data has been analysed later applying the 3 different models? This would be important to know for the later statistical analysis.

DISCUSSION TO ISSUE 2.8

The spectra had been acquired once with the procedure described in lines 150-152. The models provided in real time the three MC estimates (integrated in the manuscript in lines 173-174).

2.9. Line 156 and 160: Different sample numbers are given here than in Line 110. Where does these samples come from? What kind of material was used here? Are these just sub-samples from the earlier mentioned 817 samples?

DISCUSSION TO ISSUE 2.9

As mentioned in lines 175-179 of the original manuscript, the samples used for model building were a different set of industrial wood chips. We integrated the description of these datasets of samples (lines 185-195).

2.10. Line 157: MC, is it wet or dry based?

DISCUSSION TO ISSUE 2.10

That's a good point. It should be always clarified when dealing with MC of wood/biomass. In our case we are considering MC on wet basis as this is the common "language" of our biomass users. We specified it on line 131.

2.11. Fig. 3 Units at the x/y axis are missing. Is the error in %MC or in %of the total?

DISCUSSION TO ISSUE 2.11

Also a good point! The units had been added to the figure (now Figure 4) as well as the type of average error we are talking about: the % of the bias over the measured MC.

2.12. Line 281: Other-non portable technologies: Please deliver more information about what technologies do you mean here and provide more information about the specific differences (e.g. precise numbers). Otherwise, this discussion sounds quite speculative.

DISCUSSION TO ISSUE 2.12

We integrated the sentence with the specific technologies involved (line 319).

2.13. Fig. 4 It looks like if there are some letters missing at the right side of the legends due to the cutting of the images.

DISCUSSION TO ISSUE 2.13

Indeed the legend had been cut off. We revised the original images and loaded new ones with the complete legend (now figure 5).

2.14. Please revise and be more specific. What do you mean with very dry or very wet samples (Line 338)? A range should be given here. Finally, the influence of different raw materials is not clear. Does it work with all types and qualities of wood (including bark, needles and impurities such as sand)? What could be further limitations? Please elaborate this a bit more.

DISCUSSION TO ISSUE 2.14

We detailed the extremes for very wet and very dry MC (line 378). Conclusions had been integrated with further analysis of potential limitations and need of further research to address the possible influence of impurities and/or specific assortments such as bark, leaves or needles.

Reviewer 3

3.

3.1. I suggest to add one sentence providing a strong rationale for the study, not only based on its application but in the fact that has not been yet done by previous authors (not at least in this detail).

DISCUSSION TO ISSUE 3.1

Thanks for the suggestion. We added a brief text highlighting this aspect (lines 97-98)

3.2. L45 and on. (1st P). The paragraph seems long in excess. L54 cut be the start of a new paragraph, as well as L66.

DISCUSSION TO ISSUE 3.2

Thanks for revising this detail. The manuscript had been modified accordingly.

3.3. L125 The integration time...

DISCUSSION TO ISSUE 3.3

Corrected as suggested.

3.4. L130 replicates For... (something is missing)

DISCUSSION TO ISSUE 3.4

Thanks for identifying this error. Writing and rethinking a manuscript always generates a plethora of such incomplete or truncated sentences. The sentence had been revised and corrected.

3.5. L138 Precision test (?) Precision assessment (?)

DISCUSSION TO ISSUE 3.5

We like the first proposal. This section had been renamed “Precision test”

3.6. NIR (acronym) should be properly defined in all captions at tables/figs. Same applies to R&R (fig5 etc...). In the captions, many capital letters are not required (e.g., "for Deviation values" > "for deviation values"). Same for tables (e.g., see Table 4)

DISCUSSION TO ISSUE 3.6

Please apologize, we did not fully understand this point. NIR acronym had been explained in the introduction, and in the rest of the manuscript we just use the acronym to reduce the length of the text or the captions. On the contrary, we missed the explanation of the acronym R&R, so we added it at its first occurrence (line 235). We also removed as suggested all the unnecessary capital letters from the captions of figures and tables.

3.7. L153 the analysis use, in general, standard statistical methods, and I doubt about the to specify the vendor (MatLab)

DISCUSSION TO ISSUE 3.7:
The vendor had been removed as suggested.

3.8. L158 perhaps would need a more clear explanation of the transformations used at each model

DISCUSSION TO ISSUE 3.8:
We considered that the main aim of the manuscript is the comparison among the models implemented, relating the MC prediction in real application. For this reason, we opted for not detailing too much the models' development. Yet the suggestion makes sense indeed, and we opted for a compromise providing additional information about the models' features in lines 189-190 and 194-195.

3.9. L168 perhaps the authors could include estimators such as RMSD and similar for a full assessment of the deviations between both estimates?

DISCUSSION TO ISSUE 3.9:
As explained in 3.8 issue, the principal aim is to compare the three models in terms of prediction performance. For this reason, authors have decided to focus on the differences between predicted moisture content value and the one obtained from standard analysis.

3.10. L240 three decimals is enough

DISCUSSION TO ISSUE 3.10
Thanks for the suggestion. The figures had been modified accordingly.

3.11. *MOD1 and MOD2 are heteroscedastic in the predictions, with a larger predictive error in high MC. MOD3 seems to not show this trend but at the expense of a lack of linearity in the predictions (concave, versus convex in MOD2). I wonder if non-linear models (adding a correction factor for large or very small MC) could be of help here, and would avoid the proposed solution (L334).* In addition, would be beneficial to combine the methods for the estimates used in MOD2 and MOD3 (some weighted average?) to increase the precision? The range 30-50 is critical, as the authors stress. MOD3 seems to deliver more precise predictions in the range 40-50 and MOD2 in the range 30-40.
This must be the result of the transformations applied to the spectra, that work in opposite directions due to the role of the SNV in correcting the estimates. Maybe the authors could play with this, transforming SNV, adding a correcting factor at this stage?).

These are mere suggestions: I understand that the more complexity is added in the model or in the previous steps in the data analysis, the more the risk of over-fitting.

DISCUSSION TO ISSUE 3.11:
The main goal of this manuscript would like to verify the practical application of implemented prediction models on laboratory analysis and to test if performances could greatly differ. As shown, optimal results but also limited conditions concerned each model. The discussion proposed

by the revisor is very interesting to overcome the need to operator choice. Further solutions could be considered, so as suggested, a test on MOD2 data have been developed to apply different correction factors in extreme MC values. Results little improved (from 0.8814 to 0.8905) and better correlation has been shown with “stronger” correction (correcting range 10-20 and 50-70%). The non-linearity models could be related to the lack of adequate number of samples corresponding to extreme values that could be led to better performances. Further studies could be performed to add samples with higher and lower MC at the present dataset and verify the results. For this reason, a comment has been added at lines 303-306.

3.12. Supplementary data: it is misleading, as the document attached is the captions of the figures.

DISCUSSION TO ISSUE 3.12

Thanks for highlighting this issue. During the upload of the manuscript and the related files we did not find a specific item for captions in the flag menu. We upload the revised material using a different identifier.

HIGHLIGHTS

- A portable NIR spectroscope was used to estimate moisture content of biomass fuels
- Validity, accuracy and precision of 3 prediction models were compared
- Moisture estimate with portable NIR is reliable, fast and non-destructive
- Results of the prediction models differed mostly on the extreme moisture content values
- NIR spectroscopy is highly suitable to analyze fuel quality along the supply chain

Performance of a portable NIR spectrometer for the determination of moisture content of industrial wood chips fuel

Giuseppe Toscano[1]

Elena Leoni[1]

Thomas Gasperini[1]

Gianni Picchi[2*]

[1] Department of Agricultural, Food and Environmental Sciences, Università Politecnica delle Marche, via Brecce Bianche, 60131 Ancona, Italy;

[2] CNR-IBE, via Madonna del Piano 10, 50019, Sesto Fiorentino, Italy;

* corresponding author

Keywords:

NIR, moisture content, fuel quality, wood chips, bioenergy, biomass

ABSTRACT

The environmental policy of the European Union is boosting the development of renewable energies. Among these, bioenergy holds the main share and is expected to further increase. Such development requires a higher degree of efficiency in the whole supply chain. This is achieved also with an enhanced fuel quality control and a better matching with the energy conversion systems. For solid biofuels, moisture content is the main quality parameters, influencing the sustainability of the whole energy system. With the aim to provide a real-time and portable tool for moisture measurement, a hand-held near infrared spectrometer was tested on a dataset of 817 wood chip samples provided by an industrial facility. A set of key performance parameters were used to compare the estimation of three alternative prediction models and the standard oven dry method. Results show a satisfactory reliability with R^2 ranging from 0.86 to 0.89 depending on the model. A single measure can be acquired in few seconds, and the potential to deploy the non-destructive analysis directly at the fuel storage (yard) and at different steps of the supply chain discloses a wide range of options to efficiently control fuel quality.

Nomenclature and acronyms

EU	European Union
KPI	Key Performance Indicator
LVF	Linear-variable Filter
MC	Moisture Content
MOD	Model
NIR	Near InfraRed Spectroscopy
PLS	Partial Least Square regression
RE	Renewable Energy
RMSEP	Root mean square error of prediction
R&R	Repeatibility and Reproducibility test
SEP	Standard error of performance
SNV	Standard normal variate

1. Introduction

The recent European Green Deal climate actions boosted the efforts to reduce the emission of climate-altering pollutants in the European Union (EU). In particular, the "Fit for 55" package sets a maximum emission threshold to be met by 2030, corresponding to 55% of the figures recorded in 1990. This program involves particularly the energy sector, which must increase the share of renewable energy (RE) to 40% in the same time span [1]. A rather ambitious target considering that by 2017 RE provided just 17.6% of the total energy supply in the EU [2]. Since bioenergy was responsible for over 58.5% of total RE output the present stimulus is expected to increase up to fivefold the energy consumption of biomass in the next decades, strengthening its role of RE backbone in the energy mix of the EU [3]. In order to meet the expectations, the bioenergy sector must seek for a higher degree of efficiency of the whole supply chain.

This requires, among other aspects, a higher quality control of the fuel and a better matching between fuel properties and energy conversion systems. For solid biofuels, moisture content (MC) is considered as the most relevant quality factor [4] and a thorough monitoring of MC is the most cost-effective strategy for managing biofuel procurement in energy facilities, in spite of the investment in time and resources that it requires [5]. In fact, a high MC has detrimental effects on the whole forest-energy supply chain, beginning with the reduction of the effective payload of trucks, which decreases the environmental and economic sustainability of biomass procurement [6,7]. Once in the yard, long-term storage of wood chips with high MC may lead to important biomass losses due to microbial development [8–10], causing an immediate value loss and an undesired proliferation of fungal spores in the biomass piles [11]. In some cases, this process can even lead to self-ignition [12], with total destruction of the stored fuel. As a further inconvenience, a high MC strongly reduces the heating value [13], increasing the biomass required for the same energy output. Additionally, when stored in open yards the biomass is exposed to uncontrollable factors (e.g. rain, wind, etc.) that generally lead to MC increase [14,15], but may also dry the stocked fuel if the conditions are favorable [16].

This represents a further challenge as the combustion of biomass with unknown and variable MC leads to unstable and inefficient firing process, with temperature fluctuations into the furnace that may cause slagging as well as increased emissions of NOx, CO and PM [17]. This issue can be partially coped with indirect systems

for monitoring and adjusting the combustion performance, based on flue gas analysis [18] or energy output monitoring [19]. Yet, these systems based on post-combustion parameters are hindered by unavoidable inertia of reaction, which increases with the size of the furnace. In-line and real-time monitoring of the fuel fed to the furnace would be a much more effective solution to adjust combustion settings according to biomass quality. For instance, encouraging results had been obtained for in-line detection of MC with microwave reflection sensors on sawdust [20]. Another promising technology for fast determination of MC along the biomass supply chain is near infrared spectroscopy (NIR). It has already proved its potential in characterizing solid fuels on conveyor belt (in-line) [21], laboratory MC analysis [22] as well as when deployed directly in the field with portable instruments [23,24]. Overall, NIR technology proved to be reliable in the MC estimate of biofuels such as wood chips [25],pellets [26], and herbaceous biomass such as bamboo [27]. Additionally, NIR sensors can provide a wider range of services besides pure MC determination, deploying the same spectra for quantification of other fuel properties such as calorific value, ash content [28], the type of woody biomass (broadleaf or conifer) [29] or detecting the presence of herbaceous biomass [30]. In addition, the availability of portable NIR sensors with real-time measurement, allows to assess the relevant quality parameters and their spatial patterns directly on the pile or the truckload [31]. This application could strongly improve the MC control of loose industrial biofuels, as the present biomass sampling procedures struggle to achieve a compromise between reliability and acceptable costs [32,33]. An issue particularly relevant in regions with a high variety of woody biomass sources, such as Southern Europe, where these fuels feature very inhomogeneous characteristics [34–37], leading to an additional effort to control the quality of biomass feedstock.

Finally, the availability of reliable models for the determination of MC (and other quality parameters) with NIR sensors would pave the way to several applications falling in the frame of the forthcoming digitalized bioeconomy. As an example, if installed on wood chippers it would provide real-time information on fuel quality as currently is done with grain harvesters [38]. Deployed at different steps of the supply chain the sensor could monitor the quality changes of the produced and stored biomass as well as enhance fuel combustion if operated at the furnace inlet. Such development requires the identification of adequate hardware solutions and the development of reliable prediction models to convert the raw spectra in MC figures.

However, in spite of the relevance of this technology for the bioenergy sector, no research has yet focused on the deployment of hand-held, self-powered NIR sensors for the estimate of the MC of solid biofuels.

Considering the above, the present study aimed to test the performance of a portable NIR spectrometer running three different MC prediction models, assessing its potential to determine fuel quality with heterogeneous industrial biomass. Quality assurance was based on three key performance indicators (KPI) according to the guidelines suggested by Vardeman and Jobe [39]:

- "Validity" is intended as the capacity to provide results that reliably represent the quantity measured, without the influence of non-controlled factors. In this case, these would be mainly tree species and woody assortments used for the production of the wood chips. Yet, due to the lack of information regarding the biomass composition, the unique factor considered to assess the validity of estimate was the influence of the extreme MC values;

- "Accuracy", accomplished when the average of values estimated produces the true or correct values of MC as measured with the reference method;

- "Precision" related to the range of variation observed measuring samples with the same or similar MC values, which should ideally result in minimum variations;

Considering the industrial focus of the test, an additional KPI was included in the study:

- "Performance" of the analyzer, intended as the effective output of MC estimates per work hour in real work conditions.

2. Materials and methods

2.1 Wood chip samples collection and preparation

The wood chip samples (n = 817) had been provided by a power plant located in Northern Italy from July to October 2020. The facility has an electricity output of 11.6 MW and consumes about 130,000 t/year. It is fed with a wide range of fuels, including energy crops (medium rotation coppice of poplar clones), agricultural

residues (mainly from uprooting of pear, apple and peach orchards), river banks maintenance (providing a mix of broadleaves dominated by willow, poplar and alder) and conifers from a large windthrown area of the Italian Alps (mainly spruce). Biomass samples were collected in the yard of the power plant according to the sampling procedure defined by the technical standard ISO 18135:2017 – Sampling of solid biofuels [40]. The information regarding the type of biomass was available just for few samples. Additionally, this data was visually assessed by the facility's yard manager on the pile of wood chips, with a high degree of approximation. For these reasons, this descriptive data was not considered in the following analysis.

Wood chip samples with an approximate fresh weight of 500 g were delivered to the lab in hermetically sealed plastic bags to preserve their original characteristics. Here they were prepared according to the technical standard ISO 18134-1:2015 [41] for the evaluation of MC (always considered on wet basis).

2.2 Near-infrared data acquisition

NIR analysis was performed in laboratory by means of a portable MicroNIR™ OnSite sensor (Viavi Solutions Inc., Santa Rosa, CA, USA). Featuring no moving parts it can be regarded as a "rugged" hand-held spectrometer with high resistance to vibrations, dust and water (IP67). Portability is facilitated by its small size (4.6 cm of diameter and 19 cm of length), low weight (<300 g) and high battery run time (>10 hours). The model used is interfaced with a rugged tablet via USB cable (Figure 1), but the same sensor is also available with wireless Bluetooth® connection. Both models have a cost of about 10,000 €.

The instrument works in the spectral range between 950 and 1650 nm. It is equipped with two small vacuum tungsten lamps (ø ≈ 4 mm) as radiation source. Dispersion is performed by a linear-variable filter (LVF) directly connected to a 128-pixel indium gallium arsenide (InGaAs) photodiode array detector. The acquisition was carried out in reflectance mode. The integration time was 6.7 ms and each spectrum was the average of 100 scans, thus with an acquisition time below 1 second. In order to remove the instrumental and environmental noise, a dark reference (0% transmittance) and a blank spectrum have been acquired every hour using a 99% reflectance reference standard (Spectralon). All spectra were collected by operating the sensor at a stable internal temperature (30 ± 1 °C).

MC of samples was estimated as the average value of ten measurements (replicates). For this purpose, the sample was carefully distributed on an aluminum tray (30x24x4 cm), creating a uniform layer with a depth of about 2-2.5 cm . Here the NIR raw data was acquired on a matrix of 9 predefined spots plus a randomly-selected position as depicted in figure 2. The operation was performed manually by a the same operator throughout the whole study. The sensor was placed directly in contact with the surface of the sample. A perpendicular position was chosen in order to minimize light interferences and enhance the quality of acquisition. Each spot covered an area of about 250 mm^2. After spectra acquisition the sample was oven-dried for MC measurement according to standard ISO 18134-1:2015 [41].

Figure 1: portable MicroNIR™ OnSite sensor application on industrial wood chip pile in power plants (left) and in laboratory (right).

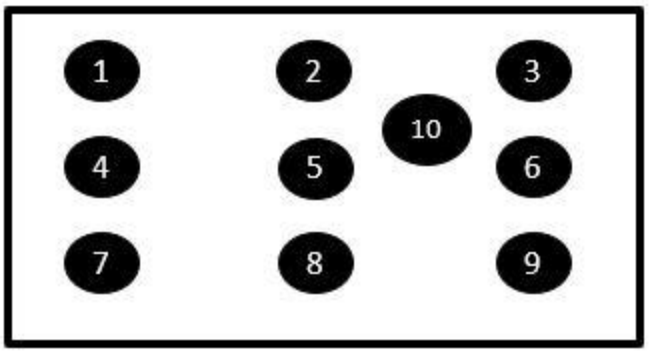


Figure 2: 3x3 matrix used for sampling NIR scans on biomass distributed on aluminium tray.

2.3 Precision test

The precision test aims at assessing the dispersion of measured values. Standard deviation is a good indicator of this performance, yet a more detailed analysis requires repeated measurements performed on a same group of samples. Therefore, 30 new wood chip samples provided by the same power plant were used to generate a dedicated dataset at the end of the main study. The NIR analysis was repeated 5 times on each sample following the protocol previously described in 2.2. Between each repeated measurement the biomass in the tray was carefully mixed. Finally, the reference MC of the biomass was determined by means of the oven-drying method (ISO 18134-1:2015).

2.4 Prediction models

The spectra acquired on the biomass samples were used to estimate MC by means of three different prediction models installed on the tablet and run simultaneously at each raw data acquisition providing real-time results. The models had been previously developed using the spectra acquired on different sets of industrial wood chip samples provided by two Italian power plants during the routine control of MC of the incoming feedstock. Although the specific characteristics of the biomass samples were unknown (e.g. tree species, storage time and conditions, etc.) a wide variability was expected, allowing for the development of robust models. Additionally, one of these power plants was the same that provided later the 817 samples used for this study. Thus the datasets deployed to build the models and to perform the analysis were considered similar in terms of fuel composition and MC variability.

All the computations have been performed in Matlab environment using in-house functions on existing algorithms. Each prediction model was selected as the best performing of a series of models computed on the averaged matrices with different pretreatments. The first prediction model (MOD1) was developed on the spectra of 642 samples with a range of MC values between 4.3% and 49.1%. A Partial Least Square regression (PLS) model was used pretreating the spectra with the combination of first derivative (Savitzky-Golay filter, 5-points window, second-order polynomial) and Standard Normal Variate (SNV). The resulting

189 model features R^2=0.94 and RMSEP=2.40%, while RER and RPD values report respectively 23.5 and 4.1,
190 confirming that it could be considered as a reliable model. The second (MOD2) and the third (MOD3)
191 prediction models have been developed on the spectra of 212 additional samples (different than the previous
192 642 samples) with a range of MC values between 15.2% and 64.7%. MOD2 was developed pretreating the
193 spectra with the second derivative (Savitzky-Golay filter, 5-points window, second-order polynomial)
194 featuring R^2=0.96, RMSEP=1.99%, RER=24.9 and RPD=4.5. MOD3 was developed as the previous one, with
195 additional SNV pretreatments resulting in R^2=0.94, RMSEP=2.44%, RER=19.7 and RPD=3.8. The RMSEP values
196 of the three models do not show significant differences among each other and are in line or superior to the
197 results of other researches estimating MC with NIR spectroscopy in woody materials [42,43] and other
198 biomasses [44,45].

199

200 2.5 Data analysis

201 To analyze the accuracy of the NIR analysis and the three models tested, the difference (bias) between the MC
202 returned by the estimate (MC_nir) and the reference value (MC_ref) was calculated as follows:

203 $$\text{Bias} = \text{MC\_nir}_{im} - \text{MC\_ref}_{i}$$

204 Where MC_nir is the value returned by the model *m* for the sample i and MC_ref is the value measured with
205 oven dry method for the same sample *i*.

206 Bias values were first checked with descriptive analysis (Box-Plot) for possible outliers (difference > 1.5 SD).
207 The first round identified a large number of anomalous values: 87, 93 and 52 respectively for MOD1, MOD2
208 and MOD3. Since the number of potential outliers was large and no clear pattern or cause of outlier generation
209 could be identified, a second identification procedure was performed. This was based on the observation of
210 normal probability plots of bias values: a single outlier was identified in MOD1 (difference > 10 SD) and
211 removed from the following analysis. The resulting databases were used to assess the key performance
212 indicators (KPI) of the NIR sensor with the three prediction models as described in the following sections.

213

214 2.5.1 Validity

215 A general statistical analysis was performed to compare the performance of the three prediction models based
216 on average, standard deviation, minimum and maximum MC values. In order to better assess the validity of
217 each model according to the MC level of the sample, the dataset was divided in homogeneous moisture classes,
218 each with a range of 10 MC percentage points.

219 Additionally, the validity of the three prediction models was verified through regression analysis, assessing
220 the linearity of MC values estimated against the values returned by the standard oven-dry method.

221

222 2.5.2 Accuracy

223 This performance indicator was verified by means of two analyses:

224 - calculating the Standard Error of Performance (SEP), as described by [46], which also allows for
225 comparison of the tested NIR models with other MC analyzers:

226 $$SEP = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(e_i - \bar{e})^2}$$

227 Where N is the number of samples; $e_i = (M_{reference}\text{-}M_i)$ and M_i is the MC measured by the analyzer for the ith sample and $M_{reference}$ is
228 the MC determined for the same sample according to the standard method; $\bar{e}$ is the average of e_i.

229

230 - identifying the Statistical Tolerance Limits. For this analysis, a Shapiro-Wilk test was first performed
231 for verifying the normal distribution of the bias datasets generated by the three prediction models.
232 Since just MOD2 data showed a normal distribution, a non-parametric tolerance limit analysis was
233 performed, considering a confidence level of 90% and capturing 91.5% of population proportion.

234

235 2.5.3 Precision

236 The precision of the three models was verified by running a Gage Repeatability and Reproducibility (R&R)
237 test, ANOVA method. This technique is specifically designed for verifying the "Repeatability" and
238 "Reproducibility" of a measurement conducted with a specific gauging device (Instrument) operated by one

or more operators (Appraiser) on one or more items (Part) [39]. The three prediction models were considered in the analysis as a different Appraiser, using a single Instrument for measuring 30 Parts (biomass samples) 5 times. With such design it was possible to verify the "Repeatibility" of the analysis (i.e. variation obtained by repeating a measure with the same instrument). The "Reproducibility" of the measurement, which is the variation due to different operators, was used to highlight the difference due to the three prediction models. The ANOVA method without interaction was chosen, as it is considered more robust than the Average and Range Method against possible interactions between samples and operators.

2.5.4 Performance

The time required for the analysis was measured for each sample (tray). Since a single operator was both carrying on the MC analysis and recording the time required, the accuracy of the timing was limited, thus a common desk watch was used to note starting and stopping time of each cycle/sample.

3. Results and Discussion

The average MC of the samples according to the standard method was 37.24%. The dataset had a very wide range, including very dry (~13%) and very wet (~70%) biomass. Comparing the average MC with the corresponding values returned by the three prediction models (table 1) differences appear very limited, confirming the general reliability of NIR sensor and an apparent superiority of MOD3. Yet, individual values, such as the maximum and minimum moisture levels reported show a high degree of variability.

Table 1: General statistics to compare the MC estimate three prediction models

Value (%)	Standard Method	MOD1	MOD2	MOD3
Average	37.24	37.52	38.45	37.29
SD	8.96	8.14	8.38	8.74
Min	12.76	17.29	21.64	9.58
Max	69.31	76.61	75.18	64.18
Range	56.55	59.32	53.54	54.60

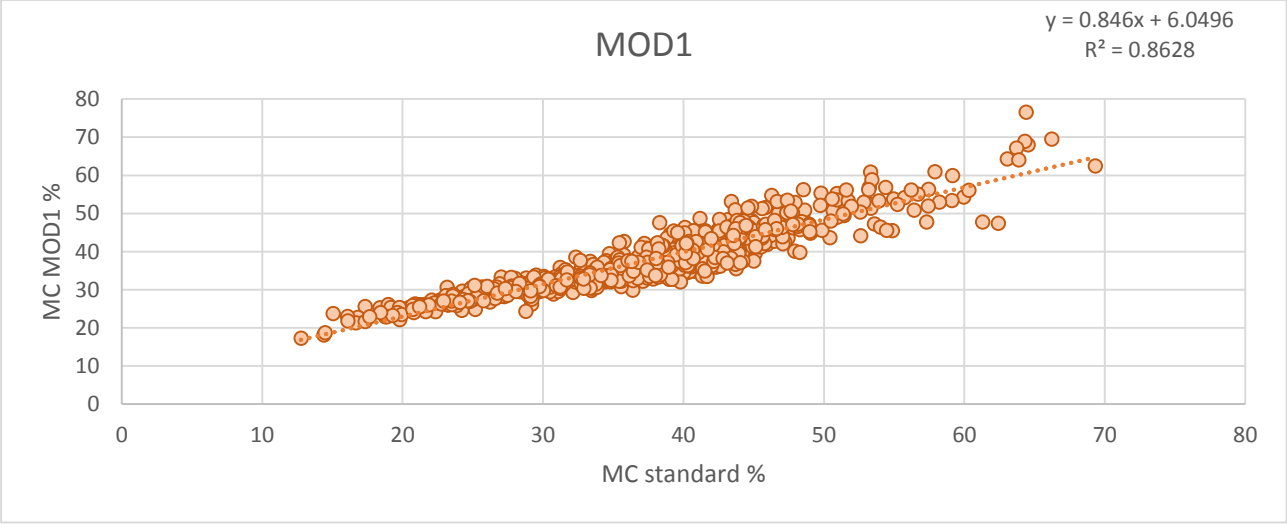
The percentage of overestimated and underestimated MC records are reported in table 2 for each prediction models. Considering absolute values, the average bias is around 2.5% for all models, while the maximum bias is produced by MOD1 (14.96%). All models show a higher frequency of overestimating occurrences compared to underestimated ones, but MOD2 is strongly asymmetric with 65.48% of estimations with a positive bias.

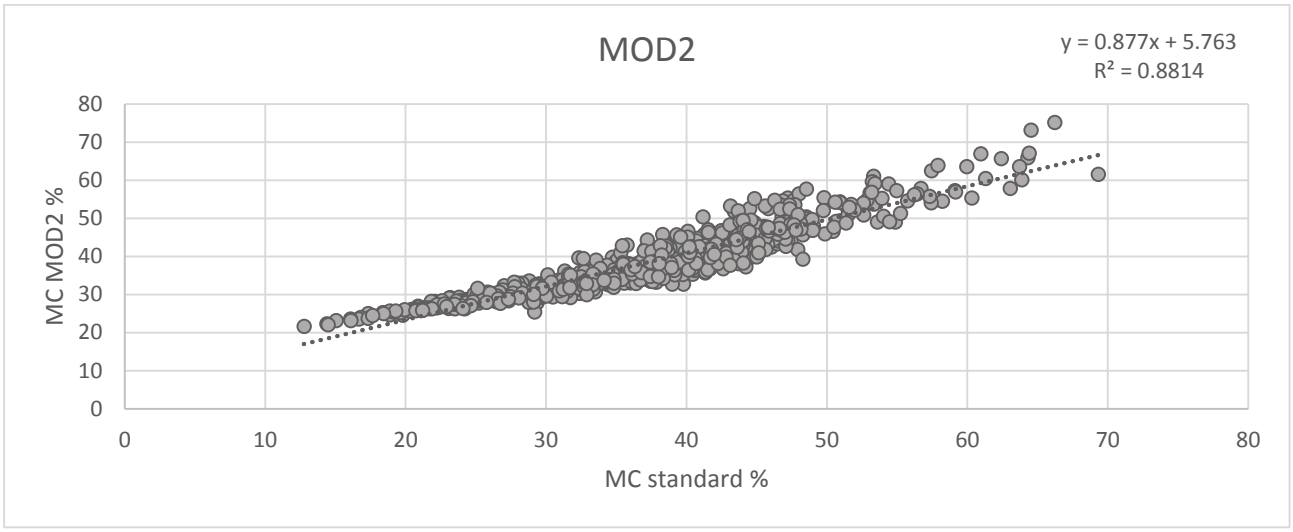
Table 2: Resulted values of bias related with each prediction model and their estimation trend

Value (%)	MOD1	MOD2	MOD3
Absolute mean bias	2,60	2,62	2,41
Max bias	14.96	10.32	11.79
Min bias	0.01	0.01	0.00
Overestimated	55.08	65.48	54.59
Underestimated	44.92	34.52	45.41

3.1 Validity

Considering the regressions of the three prediction models, the estimation capacity is satisfactory, with coefficient of determination values (R^2) ranging from 0.86 to 0.88 (figure 3). Yet, this performance is inferior to that achieved with a NIR sensor on homogeneous pelletized biomass [47] and even to that of a magnetic resonance sensor tested with samples featuring a similar variability to the present study [48]. The great variability was also influenced by the lack of explicit information about biofuel origin and tree species correspondence, that made harder the investigation on additional classification and MC influence. The bias in linearity confirms the presence of some disturbance in MC estimation, with MOD3 showing the minimum deviation from linearity ($\beta = 0.918$) to a maximum in MOD1 ($\beta = 0.846$).





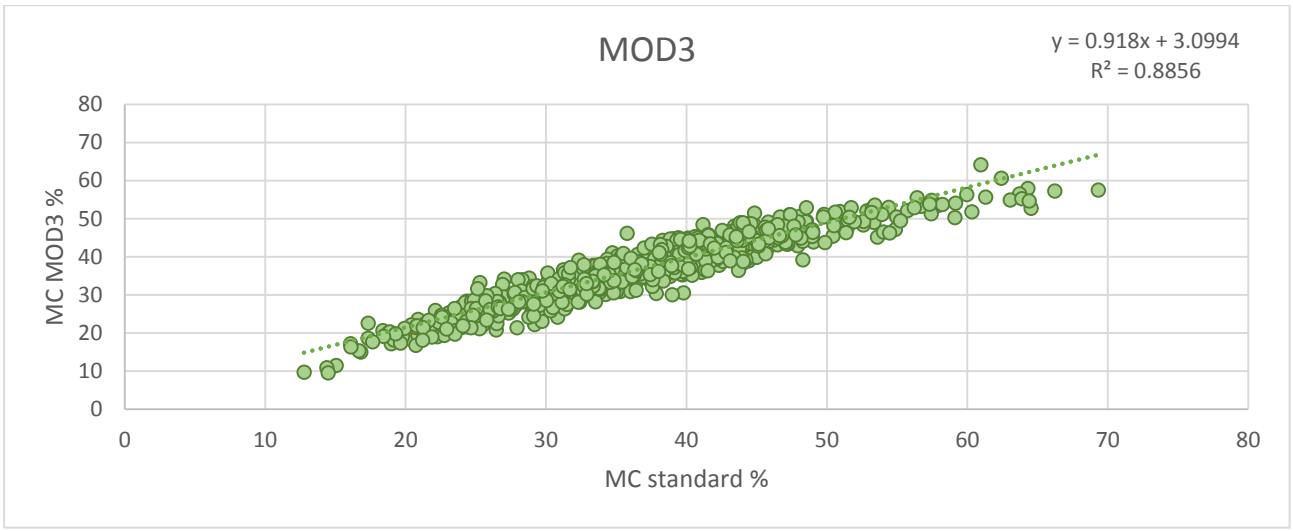


Figure 3: Relation of MC values estimated by the NIR sensor for MOD1, MOD2 and MOD3 as compared to MC estimated with standard method.

Before considering the influence of MC classes on the estimation bias returned by the models it is important to notice how the frequency of samples in each class is strongly unbalanced. As shown in table 3, 73.7% of samples are included in the two middle classes, with moisture ranging between 30 and 50%. This distribution can be considered as well representative of the actual biomass fuel used by the power plants, where extreme values tend to be exceptions.

Table 3: distribution of samples according to the MC classes

MC class	10-20	20-30	30-40	40-50	50-60	60-70
Samples (n)	23	138	353	249	42	12
Samples (%)	2.8	16.9	43.2	30.5	5.1	1.5
Avg. MC (%)	17.58	25.62	35.55	43.8	54.02	63.69

SD	1.96	2.80	2.88	2.61	2.67	2.35

Although the general performance of the models is similar when the whole dataset is considered, its validity has a different pattern when individual MC classes are considered (figure 4).

MOD1 has satisfactory reliability just for the two central MC classes, with an average bias below 0.5%, while strongly overestimates drier classes and features a very high variability when analyzing samples falling in the two classes with higher MC.

MOD2 highly overestimates (over 3%) for MC content lower than 30%, while shows a high reliability for all other MC levels, with a maximum average bias below 0.7%.

MOD3 has a reverse bias pattern compared to the previous one, with high reliability for lower and middle MC classes and strong underestimation for the two classes with higher MC.

Considering the relative weight of each class (i.e. the percentage of samples falling in it), MOD3 results to be the most reliable with 93.4% of estimates with an average bias lower than 0.7%, while MOD2 achieves 80.3% of estimates within this threshold and MOD1 just 73.7%.

Given the above considerations, MOD3 appears to provide the best performance, even if its capacity to predict MC of biomass is limited to wood chips with MC lower than 50%. Above this threshold, the analysis would return a result strongly underestimated. As several models can be run simultaneously, a practical solution to this issue could be to deploy in parallel two models for spectra interpretation: MOD3 could be used as default model, but for MC values >50% the estimated value of MOD2 could be considered since it has much higher reliability with high MC levels, and a similar one with average MC values. As an alternative, a correction factor could be applied to adjust the result of the models in extreme MC classes. Thus, a preliminary test has been performed correcting with different factors the values predicted by MOD2 resulting in a minimal correlation improvement (from 0.88 to 0.89).

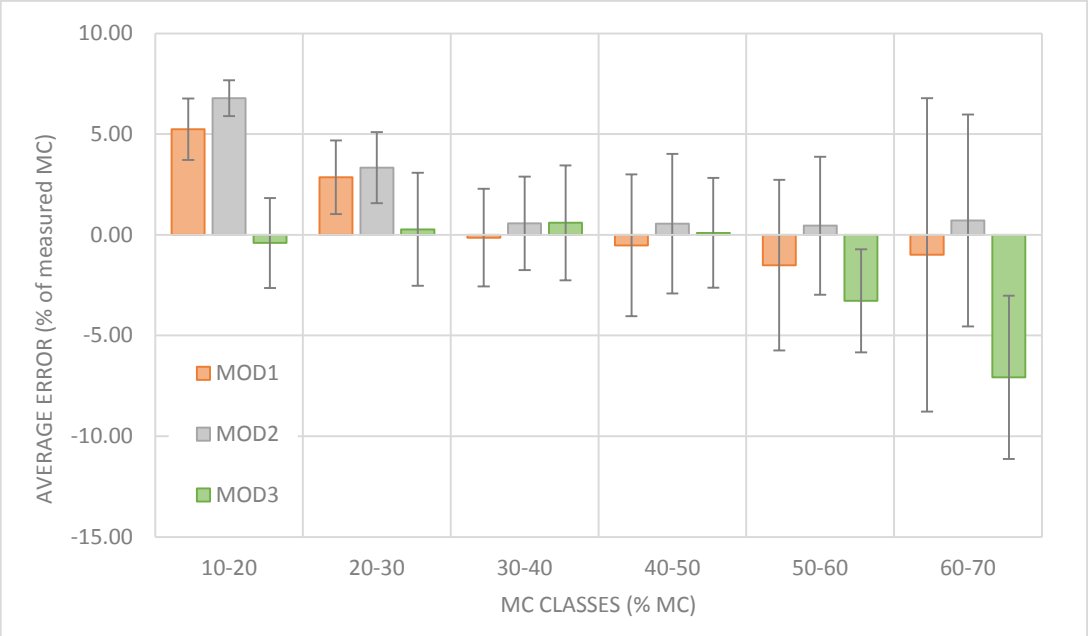
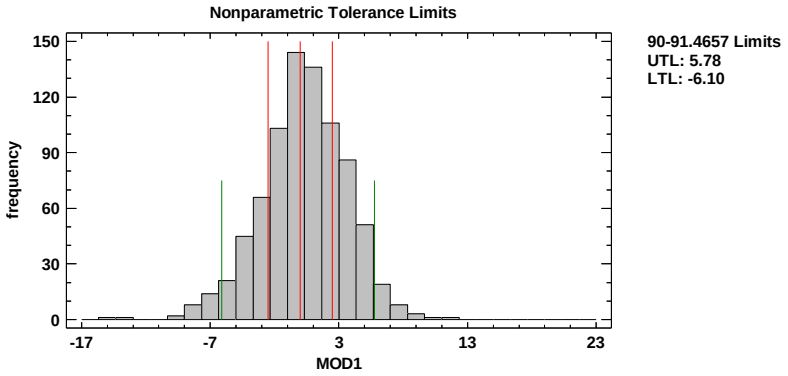


Figure 4: average error of estimate according to MC classes (as measured with standard method). Vertical bars represent the standard deviation.

3.2 Accuracy

The 30 samples used for accuracy determination had MC values ranging from 1 to 52%, thus covering most of the MC classes featured by the main database. The SEP values for the NIR sensor were 3.5%, 3.1% and 3.0% respectively for MOD1, MOD2 and MOD3. These values are in line with the average SEP reported for moisture meters based on NIR and Radio Frequency technologies, and are even similar to the SEP of the oven-dry method operated with 100g samples [46]. Other non-portable technologies for MC estimation recently tested achieved lower SEP values either deploying microwave reflection operated in-line in laboratory conditions [20] or with magnetic resonance coupled with traditional sampling in industrial environment [48]. Yet, being fixed equipment those provide a lower sampling flexibility.

Without assuming any particular distribution of the bias values, and with a confidence level of 90%, the statistical tolerance limits analysis reports that at least 91.5% of the distribution lies between limits with a span of 11.88, 11.13 and 10.54 percentage points respectively for MOD1, MOD2 and MOD3 (figure 5). This result further confirms the higher accuracy of MOD3, which also features a mean value of 0.05 against 0.32 and 1.2 respectively for MOD1 and MOD2.



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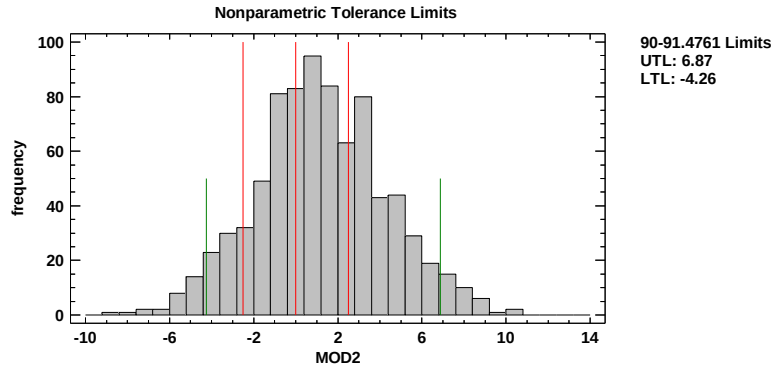

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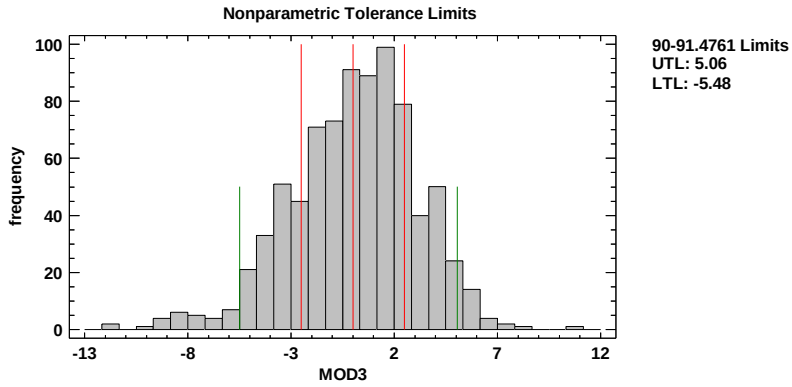

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331 Figure 5: Tolerance plot for nonparametric statistical tolerance limits. Green lines report the limits where 91.5% of
332 observation lie. Red lines represent the desired value (0, central line) and the threshold set (±2.5 points, left and right
333 lines)

334

335 3.3 Precision

336 The percent of total variation due to R&R is 24.87% (table 4). Although a threshold of 10% is generally
337 recommended (in automotive industry measurements), in other conditions with higher expected variability,
338 values within 30% are still considered acceptable. This is surely true for MC estimation of biomass where a
339 plethora of uncontrollable factors contribute to reducing the degree of both reproducibility and repeatability of
340 a measurement. The value achieved is comparable to what Aminti et al. [48] reported while assessing the
341 influence of calibration on the repeatability of a magnetic resonance MC analyzer.

Table 4: Gage repeatability and reproducibility report

Measurement Unit	Estimated Sigma	Percent Total Variation	Estimated Variance	Percent Contribution	Percent of R&R
Repeatability	2.98922	23.8036	8.93545	5.66612	**91.63**
Reproducibility	0.903729	7.19653	0.816726	0.5179	**8.37**
R & R	**3.12285**	**24.8677**	9.75218	6.18402	100.00
Parts	12.1634	96.8586	147.947	93.816	
Total Variation	**12.5579**	100.0	157.7		

In the frame of this study, the R&R analysis highlights the differences among the prediction models in terms of repeatability, with MOD2 appearing to be the better performing in terms of deviation from average (figure 6). Yet, just 8.37% of the total variance is due to the differences among the prediction models, being the remaining 91.63% related to the instrument. This result was partially expected, as the specific layout of the sensor deployed is designed for material more homogeneous than wood chips. In fact, wood surface roughness is a critical factor influencing the quality and consistency of the NIR spectra even if acquired on solid timber and polished wood samples [49,50]. Thus, this aspect is magnified when measuring loose, coarse material as industrial wood chips, leading to a less predictable illumination and reflection geometry which reduces the overall precision [49].

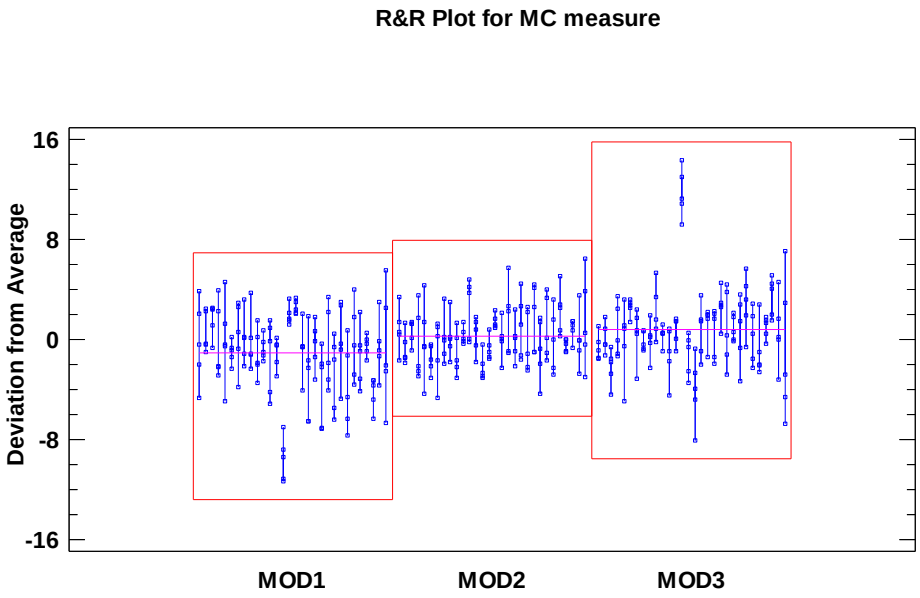

Figure 6: R&R plot for deviation values. Points represents a single measurement and are grouped by prediction model (appraiser). Horizontal red lines show the average measurement for each calibration. Vertical lines connect measurements

made on the same item: the first line in each box represents the values recorded on sample 1, the second line for sample 2 and so on.

3.4 Performance

MC measurement with the three prediction models run simultaneously (thus requiring more elaboration time) took an average of 3 seconds per spectra. A whole sample, assessed with 10 replications, could be measure in about 30 seconds. In the case of laboratory analysis, sample preparation required an additional minute to arrange the wood chips on the tray, note the ID of the sample and remove the biomass or place the tray in the oven. Overall, less than 2 minutes were sufficient to measure MC of a single sample, an analysis time comparable to that of magnetic resonance sensor [48]. Additionally, the portability of the instrument allows the operators to measure the biomass directly at the source (e.g. in the yard or on the transport unit), avoiding the collection and transfer of samples to the laboratory and minimizing the risk of sampling and laboratory errors. Finally, the real-time response of the portable sensor permits the adoption of an adaptive measurement approach [31] when deployed in situ (e.g. on piled biomass or directly on the transportation unit before unloading). A high number of sampling points appropriately selected strongly enhance the overall precision of MC estimate, making it a competitive option with the current oven-drying method, which features a much longer response time and requires a higher energy input for transportation and drying of samples.

4. Conclusions

The study demonstrated the reliability of the portable NIR sensor for the determination of MC of industrial woody fuel. Among the tested prediction models, MOD3 provides the higher level of accuracy and precision. Yet, the validity of the estimate is lower when dealing with very dry (< 15%) or very wet (> 50%) samples. This drawback is probably due to the dataset used to build the prediction models: being industrial fuel, the majority of samples belonged to the average moisture classes, reducing the power of model-training in the underrepresented extreme classes. While new models based on datasets with more homogeneous distribution of MC should be developed, the tested prediction models could be still valuable in practical application. In fact, considering the different performance of the three models at the extreme values, a higher validity could

be achieved by using the portable NIR spectrometer running two prediction models: MOD3 should be used as the main reference, but when both models return values above 50%, the result of MOD2 should be used, since this model features a higher validity at high moisture levels. The spectra acquisition is very fast, requiring about 3 seconds to return the moisture value. This performance is particularly relevant for in-field MC analysis, where the operator could gather a large quantity of spectra in a short time, reducing sampling costs and potentially applying adaptive sampling for a better estimate of the bulk quality. Future research should focus on this latter aspect, addressing the most appropriate sampling protocol for MC determination on stock piles and in transport units. Additionally, the influence of the type of raw material (e.g. tree species or tree parts) and the variability due to different operators should be further investigated in order to fully disclose the potential of this technology in the bioenergy sector.

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Performance of a portable NIR spectrometer for the determination of moisture content of industrial ~~solid biofuel~~wood chips fuel

Giuseppe Toscano[1]

Elena Leoni[1]

Thomas Gasperini[1]

Gianni Picchi[2*]

[1] Department of Agricultural, Food and Environmental Sciences, Università Politecnica delle Marche, via Brecce Bianche, 60131 Ancona, Italy;

[2] CNR-IBE, via Madonna del Piano 10, 50019, Sesto Fiorentino, Italy;

* corresponding author

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ABSTRACT

The environmental policy of the European Union is boosting the development of renewable energies. Among these, bioenergy holds the main share and is expected to further increase. Such development requires a higher degree of efficiency in the whole supply chain. This is achieved also with an enhanced fuel quality control and a better matching with the energy conversion systems. For solid biofuels, moisture content is the main quality parameters, influencing the sustainability of the whole energy system. With the aim to provide a real-time and portable tool for moisture measurement, a hand-held near infrared spectrometer was tested on a dataset of 817 ~~woodchip~~wood chip samples provided by an industrial facility. A set of key performance parameters were used to compare the estimation of three alternative prediction models and the standard oven dry method. Results show a satisfactory reliability with R^2 ranging from 0.86 to 0.89 depending on the model. A single measure can be acquired in few seconds, and the potential to deploy the non-destructive analysis directly at the fuel storage (yard) and at different steps of the supply chain discloses a wide range of options to efficiently control fuel quality.

Nomenclature and acronyms

EU	European Union
KPI	Key Performance Indicator
LVF	Linear-variable Filter
MC	Moisture Content
MOD	Model
NIR	Near InfraRed Spectroscopy
PLS	Partial Least Square regression
RE	Renewable Energy
RMSEP	Root mean square error of prediction
R&R	Repeatibility and Reproducibility test
SEP	Standard error of performance
SNV	Standard normal variate

1. Introduction

The recent European Green Deal climate actions boosted the efforts to reduce the emission of climate-altering pollutants in the European Union (EU). In particular, the "Fit for 55" package sets a maximum emission threshold to be met by 2030, corresponding to 55% of the figures recorded in 1990. This program involves particularly the energy sector, which must increase the share of renewable energy (RE) to 40% in the same time span [1]. A rather ambitious target considering that by 2017 RE provided just 17.6% of the total energy supply in the EU [2]. Since bioenergy was responsible for over 58.5% of total RE output the present stimulus is expected to increase up to fivefold the energy consumption of biomass in the next decades, strengthening its role of RE backbone in the energy mix of the EU [3]. In order to meet the expectations, the bioenergy sector must seek for a higher degree of efficiency of the whole supply chain.

This requires, among other aspects, a higher quality control of the fuel and a better matching between fuel properties and energy conversion systems. For solid biofuels, moisture content (MC) is considered as the most relevant quality factor [4] and a thorough monitoring of MC is the most cost-effective strategy for managing biofuel procurement in energy facilities, in spite of the investment in time and resources that it requires [5]. In fact, a high MC has detrimental effects on the whole forest-energy supply chain, beginning with the reduction of the effective payload of trucks, which decreases the environmental and economic sustainability of biomass procurement [6,7]. Once in the yard, long-term storage of ~~woodchips~~wood chips with high MC may lead to important biomass losses due to microbial development [8–10], causing an immediate value loss and an undesired proliferation of fungal spores in the biomass piles [11]. In some cases, this process can even lead to self-ignition [12], with total destruction of the stored fuel. As a further inconvenience, a high MC strongly reduces the heating value [13], increasing the biomass required for the same energy output. Additionally, when stor~~age~~ed in ~~uncovered yards may modify, where the biomass is exposed to uncontrollable factors such as rain, snow~~open yards the biomass is exposed to uncontrollable factors (e.g. rain, wind, etc.) that ~~and wind~~ generally lead to~~s~~ MC increase [14,15], but ~~it~~ may also ~~also reduce it~~ dry the stocked fuel if the conditions are ~~favourable~~favorable [16].

This represents a further challenge as the combustion of biomass with unknown and variable MC leads to unstable and inefficient firing process, with temperature fluctuations into the furnace that may cause slagging as well as increased emissions of NOx, CO and PM [17]. This issue can be partially coped with indirect systems for monitoring and adjusting the combustion performance, based on flue gas analysis [18] or energy output monitoring [19]. Yet, these systems based on post-combustion parameters are hindered by unavoidable inertia of reaction, which increases with the size of the furnace. In-line and real-time monitoring of the fuel fed to the furnace would be a much more effective solution to adjust combustion settings according to biomass quality. For instance, encouraging results had been obtained for in-line detection of MC with microwave reflection sensors on sawdust [20]. Another promising technology for fast determination of MC along the biomass supply chain is near infrared spectroscopy (NIR). It has already proved its potential in characterizing solid fuels on conveyor belt (in-line) [21], laboratory MC analysis [22] as well as used when deployed directly in the field with portable instruments [23,24] thanks to its compact and handheld features. MC prediction attained high and reliable performances withOverall, NIR technology proved to be reliable in the MC estimate of MC of biofuels such as applications on woody biomass, as woodchipwood chips [25], wood , but also pellets [26], and herbaceous biomass such as bamboo thanks to efficient bonds detection related to water presence that affect absorbance [27]. Additionally, NIR sensors can provide a wider range of services besides pure MC determination, deploying the same spectra for quantification of other fuel properties such as calorific value, ash content [28], and the type of woody biomass (broadleaf or conifer) [29] or detecting the presence of but also herbaceous biofuel biomass [30]. In addition, the availability of portable NIR sensors with real-time measurement, allows to assess the relevant quality parameters and their spatial patterns directly on the pile or the truckload [31]. This application could strongly improve the MC control of loose industrial biofuels, as the present biomass sampling procedures struggle to achieve a compromise between reliability and acceptable costs [32,33]. An issue particularly relevant in regions with a high variety of woody biomass sources, such as Southern Europe, where these fuels feature very inhomogeneous characteristics [34–37], leading to an additional effort to control the quality of biomass feedstock.

Finally, the availability of a portable reliable models for for the determination of MC (and other quality parameters) with NIR tool for the determination of MC (and other quality parameters) of woody biomass sensors would pave the way to several applications falling in the frame of the forthcoming digitalized

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bioeconomy. As an example, if installed on wood chippers it would provide real-time information on fuel quality as currently is done with grain harvesters [38]. Deployed at different steps of the supply chain the sensor could monitor the quality changes of the produced and stored biomass as well as enhance fuel combustion if operated at the furnace inlet. S~~Yet, s~~uch development requires the identification of adequate hardware solutions and the development of reliable prediction models to convert the raw spectra in MC figures. However, in spite of the relevance of this technology for the bioenergy sector, no research has yet focused on the deployment of hand-held, self-powered NIR sensors for the estimate of the MC of solid biofuels.

Considering the above, the present study aimed to test the performance of a portable NIR spectrometer running three different MC prediction models, assessing its potential to determine fuel quality with heterogeneous industrial biomass. Quality assurance was based on three key performance indicators (KPI) according to the guidelines suggested by Vardeman and Jobe [39]:

- "Validity" is intended as the capacity to provide results~~data~~ that reliably represent the quantity measured ~~reliably~~, without the influence of non-controlled factors ~~other than the desired ones~~. In this case, these would be mainly tree species and woody assortments used for the production of the wood chips. Yet, due to the lack of information regarding the biomass ~~quality~~composition, the unique factor considered to assess the validity of estimate was the influence of the extreme MC values;

- "Accuracy", accomplished when the average of values estimated produces the true or correct values of MC as measured with the reference method;

- "Precision" related to the range of variation observed measuring samples with the same or similar MC values, which should ideally result in minimum variations;

~~- "Accuracy", accomplished when the average of values estimated produces the true or correct values of MC as measured with the reference method;~~

Considering the industrial focus of the test, an additional KPI was included in the study:

- "Performance" of the analyzer, intended as the effective output of MC estimates per work hour in real work conditions.

2. Materials and methods

2.1 ~~Woodchip~~Wood chip samples collection and preparation

The ~~woodchip~~wood chip samples (n = 817) had been provided by a power plant located in Northern Italy from July to October 2020. The facility has an electricity output of 11.6 MW and consumes about 130,000 t/year. It is fed with ~~consumes~~ a ~~very~~ wide range of fuels, including energy crops (medium rotation coppice of poplar clones), agricultural residues (mainly from uprooting of pear, apple and peach orchards), river banks maintenance (providing a mix of broadleaves dominated by willow, poplar and alder) and conifers from a large windthrown area of the Italian Alps (mainly spruce). Biomass samples were collected in the yard of the power plant according to the sampling procedure defined by the technical standard ISO 18135:2017 – Sampling of solid biofuels [40]~~.~~. The information regarding the type of biomass was available just for few samples. Additionally, this data was visually assessed by the facility's yard manager on the pile of wood chips, with a high degree of approximation. For these reasons, this descriptive data was not considered in the following analysis.

~~Great variability among samples has been expected, but the specie and the particle size of each biofuel sample have not been considered due to the lack of available information and to the huge number of analyzed samples. Moreover, the possible presence of mixed sample type in the same truck would make the specie classification not applicable. All the~~W~~-w~~ood chip samples with an approximate fresh weight of 500 g ~~had been~~were delivered ~~directly~~ to the lab in hermetically sealed plastic bags to preserve their original characteristics. ~~In order to maintain their representativeness, biomass was collected according to the sampling procedure defined by the technical standard ISO 18135:2017 – Sampling of solid biofuels [40]. The samples~~Here they ~~had~~were ~~been~~ prepared according to the technical standard ISO 18134-1:2015 [41] for the evaluation of MC (always considered on wet basis). ~~according to the technical standard ISO 18134-1:2015 [41].~~

2.2 Near-infrared data acquisition

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NIR analysis was performed in laboratory by means of a portable MicroNIR™ OnSite sensor (Viavi Solutions Inc., Santa Rosa, CA, USA)., ~~which f~~Featuring no moving parts it can be regarded as a "rugged" hand-held spectrometer with high resistance to vibrations, dust and water (IP67)~~, improving its resistance to dust, water, and vibrations~~. P~~Its p~~ortability is ~~confirmed~~facilitated by ~~its little~~ small size (4.6 ~~m~~cm of diameter and 19~~1~~ ~~m~~cm of length), low weight (<300 g~~r~~) and ~~by~~ high battery run time (>10 hours). The model used is interfaced with a rugged tablet via USB cable (Figure 1), but the same sensor is also available with wireless Bluetooth® connection. Both models have a cost of about 10,000 €.

The instrument works in the spectral range between 950 and 1650 nm.~~,~~ ~~i~~It is equipped with two small vacuum tungsten lamps (ø ≈ 4 mm) as radiation source. Dispersion is ~~and a~~ performed by a linear-variable filter (LVF) ~~as dispersing element~~ directly connected to a 128-pixel indium gallium arsenide (InGaAs) photodiode array detector. ~~.~~ The acquisition was carried out in reflectance mode. The i~~I~~ntegration time was 6.7 ms and each spectrum was the average of 100 scans, thus with an acquisition time below 1 second. In order to remove the instrumental and environmental noise, a dark reference (0% transmittance) and a blank spectrum have been acquired every hour using a 99% reflectance reference standard (Spectralon). All spectra were collected by operating the sensor at a stable internal temperature (30 ± 1 °C).

MC of samples was estimated as the average value of ten measurements (replicates). For this purpose, the sample was carefully distributed on an aluminum~~a~~ tray (30x24x4 cm), ~~providing~~creating a uniform layer with a depth of about 2-2.5 cm . Here ~~layer to cover the tray base as much as possible, considering the heterogenous shape of samples, where~~ the NIR raw data was acquired on a matrix of 9 predefined spots plus a randomly-selected position as depicted in figure ~~1~~2. The operation was performed manually by a the same ~~unique~~ operator throughout the whole study. ~~The instrument was directly placed in contact with the sample surface to reduce as possible lights interferences, so better acquisitions were expected when a 90° angle between light direction and samples has been achieved.~~The sensor was placed directly in contact with the surface of the sample. A perpendicular position was chosen in order to minimize light interferences and enhance the quality of acquisition. Each spot covered an area of about ~~around~~ 250 mm^2. After spectra acquisition the sample was oven-dried for MC measurement according to standard ISO 18134-1:2015 [41].

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Figure 1: portable MicroNIR™ OnSite sensor application on industrial ~~woodchip~~wood chip pile in power plants (left) and in laboratory (right).

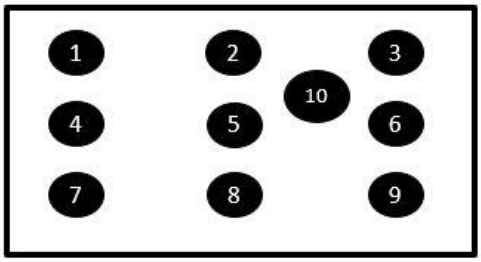

Figure ~~1~~2: 3x3 matrix used for sampling NIR scans on biomass distributed on aluminium tray. ~~on the woodchip tray (30x24x4 cm)~~

2.3 P~~Test of p~~recision test

The precision test aims at assessing the dispersion of measured values. Standard deviation is a good indicator of this performance, yet a more detailed analysis requires repeated measurements performed on a same group of samples. Therefore, 30 new ~~woodchip~~wood chip samples provided by the same power plant were used to generate a dedicated dataset at the end of the main study. The NIR analysis was repeated 5 times on each sample following the protocol previously described in 2.2. Between each repeated measurement the biomass in the tray was carefully mixed. Finally, the reference MC of the biomass was determined by means of the oven-drying method (ISO 18134-1:2015).

2.4 Prediction models

The spectra acquired on the biomass samples were used to estimate MC by means of three different prediction models installed on the tablet and run simultaneously at each raw data acquisition providing real-time results. ~~These~~ The models had been previously developed using the spectra acquired on different sets of industrial wood chip samples provided ~~to the laboratory~~ by ~~several~~ two Italian power plants during the routine control of MC of the incoming feedstock. Although the specific characteristics of the biomass samples were unknown (e.g. tree species, storage time and conditions, etc.) a wide variability was expected, allowing for the development of robust models. Additionally, one of these power plants was the same that provided later the 817 samples used for this study. Thus the datasets deployed to build the models and to perform the analysis were considered similar in terms of fuel composition and MC variability.

All the computations have been performed in Matlab environment ~~(ver. 7.10.0, The MathWorks)~~ using in-house functions on existing algorithms.

Each prediction model was selected as the best performing of a series of models computed on the averaged matrices with different pretreatments. ~~The acquisition was carried out in the reflectance mode and ten~~

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~~replicates were acquired for each sample.~~ The first prediction model (MOD1) was developed on the spectra of 642 samples with a range of MC values between 4.3% and 49.1%. A Partial Least Square ~~PLS~~ regression (PLS) model was used pretreating the spectra with the combination of first derivative (Savitzky-Golay filter, 5-points window, second-order polynomial) and Standard Normal Variate (SNV). The resulting model features R^2=0.94 and RMSEP=2.40%, while RER and RPD values report respectively 23.5 and 4.1, confirming that it could be considered as a reliable model. The second (MOD2) and the third (MOD3) prediction models have been developed on the spectra of 212 additional samples (different than the previous 642 samples) with a range of MC values between 15.2% and 64.7%. MOD2 was developed pretreating the spectra with the second derivative (Savitzky-Golay filter, 5-points window, second-order polynomial) featuring R^2=0.96 ~~and~~, RMSEP=1.99%~~, while~~ RER=24.9 and RPD=4.5. MOD3 was developed as the previous one, with additional SNV pretreatments resulting in R^2=0.94 ~~and~~ , RMSEP=2.44%, ~~while~~ RER=19.7 and RPD=3.8. ~~TFor both MOD2 and MOD3 the application reliability of prediction models was revealed. Moreover, The~~the RMSEP values of the three models do not show significant differences among each other and are in line or superior to the results of other researches estimating MC with NIR spectroscopy in woody materials [42,43] and other biomasses [44,45].

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2.5 Data analysis

To analyze the accuracy of the NIR analysis and the three models tested, the difference ~~in moisture content~~ (bias) between the MC returned by the estimate (MC_nir) and the reference value (MC_ref) was calculated as follows:

$$Bias = MC\_nir_{im} - MC\_ref_i$$

Where MC_nir is the value returned by the model *m* for the sample i and MC_ref is the value measured with oven dry method for the same sample *i*.

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Bias values were first checked with descriptive analysis (Box-Plot) for possible outliers (difference > 1.5 SD). The first round identified a large number of anomalous values: 87, 93 and 52 respectively for MOD1, MOD2

and MOD3. Since the number of potential outliers was large and no clear pattern or cause of outlier generation could be identified, a second identification procedure was performed. This was based on the observation of normal probability plots of bias values: a single outlier was identified in MOD1 (difference > 10 SD) and removed from the following analysis. The resulting databases were used to assess the key performance indicators (KPI) of the NIR sensor with the three prediction models as described in the following sections.

2.5.1 Validity

A general statistical analysis was performed to compare the performance of the three prediction models based on average, standard deviation, minimum and maximum MC values. In order to better assess the validity of each model according to the MC level of the sample, the dataset was divided in homogeneous moisture classes, each with a range of 10 MC percentage points.

Additionally, the validity of the three prediction models was verified through regression analysis, assessing the linearity of MC values estimated against the values returned by the standard oven-dry method.

2.5.2 Accuracy

This performance indicator was verified by means of two analyses:

- calculating the Standard Error of Performance (SEP), as described by [46], which also allows for comparison of the tested NIR models with other MC analyzers:

$$SEP = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(e_i - \bar{e})^2}$$

Where N is the number of samples; $e_i = (M_{reference}-M_i)$ and M_i is the MC measured by the analyzer for the ith sample and $M_{reference}$ is the MC determined for the same sample according to the standard method; $\bar{e}$ is the average of e_i.

- identifying the Statistical Tolerance Limits. For this analysis, a Shapiro-Wilk test was first performed for verifying the normal distribution of the bias datasets generated by the three prediction models.

Since just MOD2 data showed a normal distribution, a non-parametric tolerance limit analysis was performed, considering a confidence level of 90% and capturing 91.5% of population proportion.

2.5.3 Precision

The precision of the three models was verified by running a Gage Repeatability and Reproducibility ~~(~~R&R~~)~~ test, ANOVA method. This technique is specifically designed for verifying the "~~Repeatability~~Repeatability" and "Reproducibility" of a measurement conducted with a specific gauging device (Instrument) operated by one or more operators (Appraiser) on one or more items (Part) [39]. The three prediction models were considered in the analysis as a different Appraiser, using a single Instrument for measuring 30 Parts (biomass samples) 5 times. With such design it was possible to verify the "Repeatability" of the analysis (i.e. variation obtained by repeating a measure with the same instrument). The "Reproducibility" of the measurement, which is the variation due to different operators, was used to highlight the difference due to the three prediction models. The ANOVA method without interaction was chosen, as it is considered more robust than the Average and Range Method against possible interactions between samples and operators.

2.5.~~3~~4 Performance

The time required for the analysis was measured for each sample (tray). Since a single operator was both carrying on the MC analysis and recording the time required, the accuracy of the timing was limited, thus a common desk watch was used to note starting and stopping time of each cycle/sample.

3. Results and Discussion

The average MC of the samples according to the standard method was 37.24%. The dataset had a very wide range, including very dry (~13%) and very wet (~70%) biomass. Comparing the average MC with the corresponding values returned by the three prediction models (table 1) differences appear very limited, confirming the general reliability of NIR sensor and an apparent superiority of MOD3. Yet, individual values, such as the maximum and minimum moisture levels reported show a high degree of variability.

Table 1: General statistics to compare the MC estimate three prediction models

Value (%)	Standard Method	MOD1	MOD2	MOD3

Average	37.24	37.52	38.45	37.29
SD	8.96	8.14	8.38	8.74
Min	12.76	17.29	21.64	9.58
Max	69.31	76.61	75.18	64.18
Range	56.55	59.32	53.54	54.60

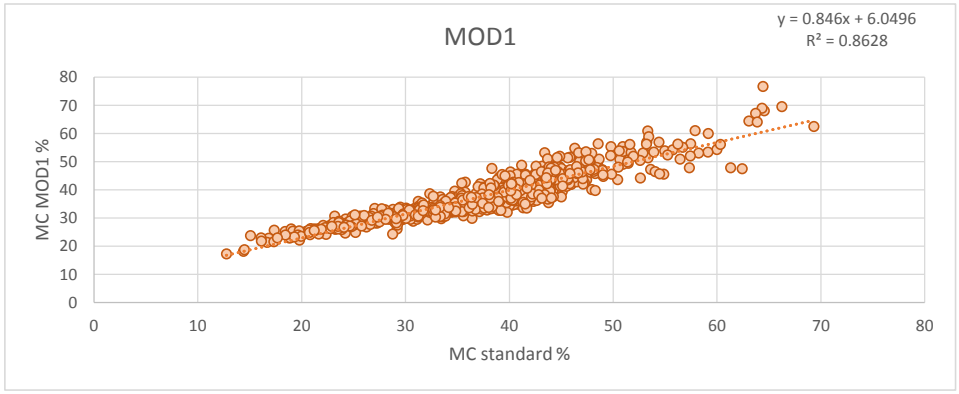
The percentage of overestimated and underestimated MC records are reported in table 2 for each prediction models. Considering absolute values, the average bias is around 2.5% for all models, while the maximum bias is produced by MOD1 (14.96%). All models show a higher frequency of overestimating occurrences compared to underestimated ones, but MOD2 is strongly asymmetric with 65.48% of estimations with a positive bias.

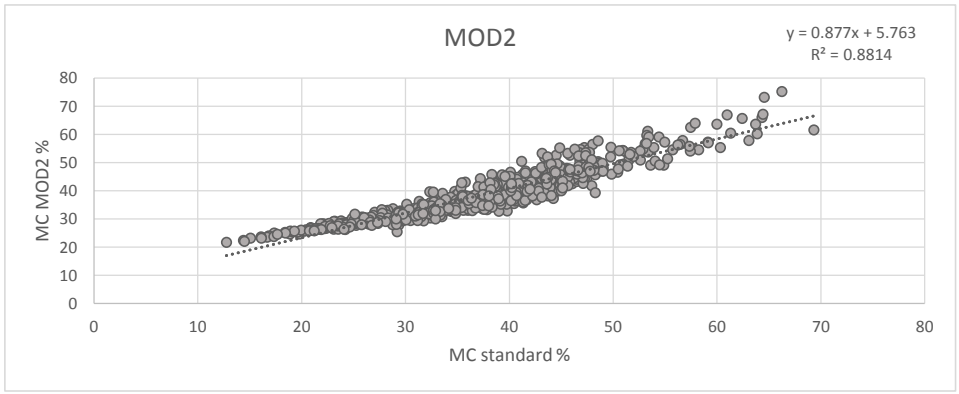
Table 2: Resulted values of bias related with each prediction model and their estimation trend

Value (%)	MOD1	MOD2	MOD3
Absolute mean bias	2,60	2,62	2,41
Max bias	14.96	10.32	11.79
Min bias	0.01	0.01	0.00
Overestimated	55.08	65.48	54.59
Underestimated	44.92	34.52	45.41

3.1 Validity

Considering the regressions of the three prediction models, the estimation capacity is satisfactory, with coefficient of determination values (R^2) ranging from 0.86 to 0.88 (figure 3~~2~~). Yet, this performance is inferior to that achieved with a NIR sensor on homogeneous pelletized biomass [47] and even to that of a magnetic resonance sensor tested with samples featuring a similar variability to the present study [48]. The great variability was also influenced by the lack of explicit information about biofuel origin and tree species correspondence, that made harder the investigation on additional classification and MC influence. The bias in linearity confirms the presence of some disturbance in MC estimation, with MOD3 showing the minimum deviation from linearity ($\beta = 0.918$~~1~~) to a maximum in MOD1 ($\beta = 0.846$~~0~~).





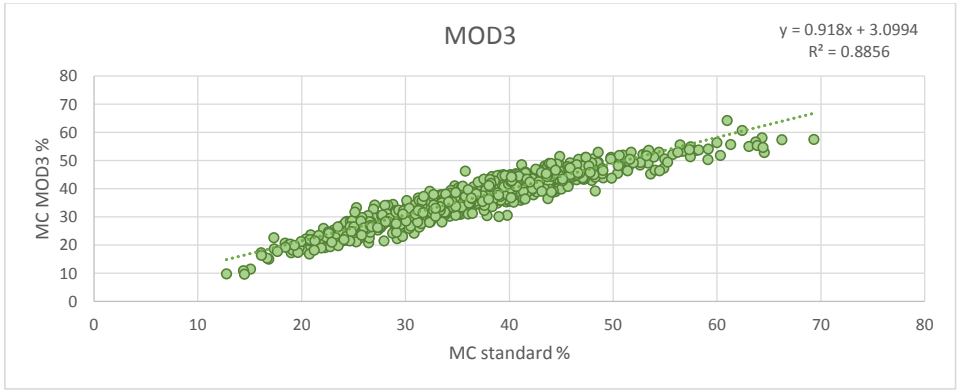


Figure ~~2~~3: Relation of MC values estimated by the NIR sensor for MOD1, MOD2 and MOD3 as compared to MC estimated with standard method.

Before considering the influence of MC classes on the estimation bias returned by the models it is important to notice how the frequency of samples in each class is strongly unbalanced. As shown in table 3, 73.7% of samples are included in the two middle classes, with moisture ranging between 30 and 50%. This distribution can be considered as well representative of the actual biomass fuel used by the power plants, where extreme values tend to be exceptions.

Table 3: distribution of samples according to the MC classes

MC class	10-20	20-30	30-40	40-50	50-60	60-70
Samples (n)	23	138	353	249	42	12
Samples (%)	2.8	16.9	43.2	30.5	5.1	1.5
Avg. MC (%)	17.58	25.62	35.55	43.8	54.02	63.69
SD	1.96	2.80	2.88	2.61	2.67	2.35

Although the general performance of the models is similar when the whole dataset is considered, its validity has a different pattern when individual MC classes are considered (figure ~~3~~4).

MOD1 has satisfactory reliability just for the two central MC classes, with an average bias below 0.5%, while strongly overestimates drier classes and features a very high variability when analyzing ~~underestimates~~ samples falling in the two classes with higher MC.

MOD2 highly overestimates (over 3%) for MC content lower than 30%, while shows a high reliability for all other MC levels, with a maximum average bias below 0.7%.

MOD3 has a reverse bias pattern compared to the previous one, with high reliability for lower and middle MC classes and strong underestimation for the two classes with higher MC.

Considering the relative weight of each class (i.e. the percentage of samples falling in it), MOD3 results to be the most reliable with 93.4% of estimates with an average bias lower than 0.7%, while MOD2 achieves 80.3% of estimates within this threshold and MOD1 just 73.7%.

~~Considering~~ Given the above considerations, MOD3 appears to provide the best performance, even if its capacity to predict MC of biomass is limited to ~~woodchips~~wood chips with MC lower than 50%. Above this threshold, the analysis would return a result strongly underestimated. As several models can be run simultaneously, a practical solution to this issue ~~w~~could be to deploy in parallel two models for spectra

interpretation: MOD3 could be used as default model, but for MC values >50% the estimated value of MOD2 could be considered since it has much higher reliability with high MC levels, and a similar one with average MC values. As an alternative, ~~Further chemometric analysis could be developed in order to avoid the necessary operator choice. A~~a correction factor could be ~~considered to verify the potential improvement of~~applied to adjust the result of the models in extreme MC classes. Thus, a preliminary test has been performed correcting with different factors the values predicted by MOD2 resulting in ~~predicted MC results~~a minimal correlation improvement ~~. A little better correlation~~ (from 0.88 to 0.89). ~~has been achieved, but stronger corrections could be required. The non-linearity models and the low improvement could be related to the lack of adequate number of samples corresponding to extreme values and the samples integration could enhance better performances.~~

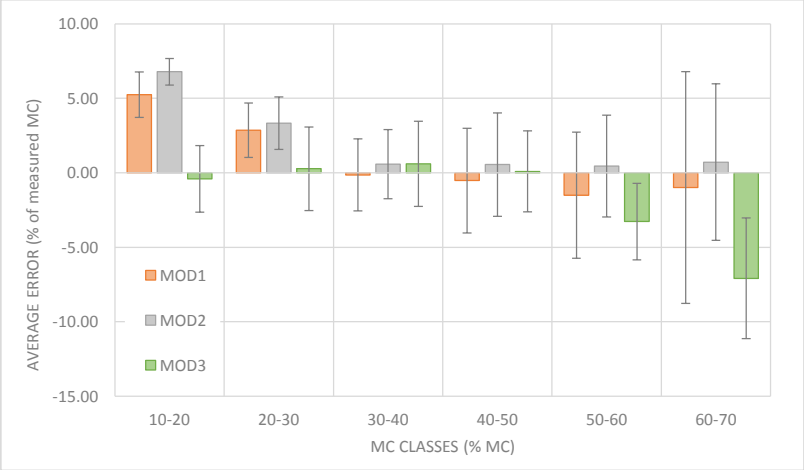


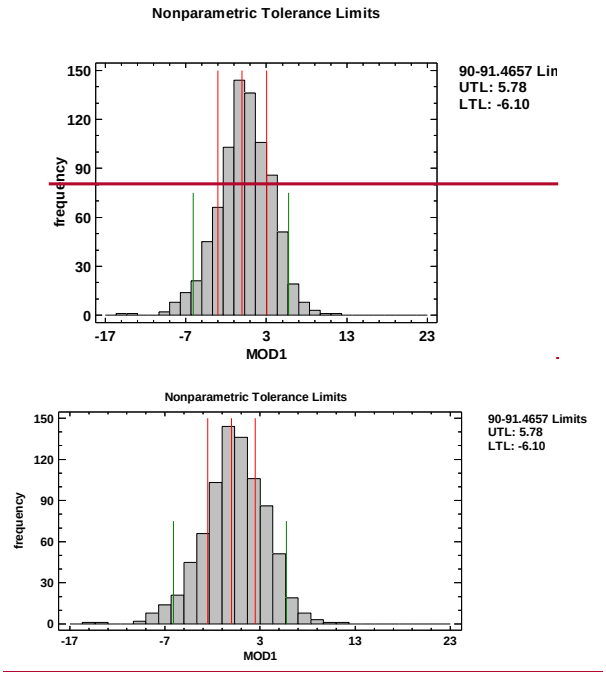
Figure ~~3~~4: average error of estimate according to MC classes (as measured with standard method). Vertical bars represent the standard deviation.

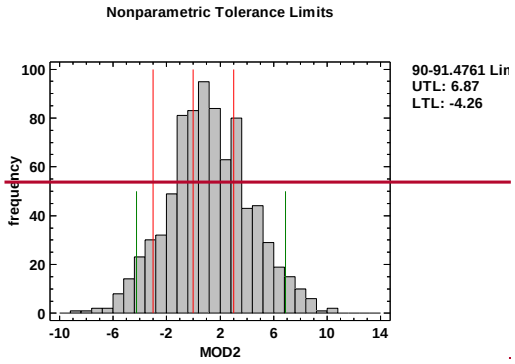
3.2 Accuracy

The 30 samples used for accuracy determination had MC values ranging from 1 to 52%, thus covering most of the MC classes featured by the main database. The SEP values for the NIR sensor were 3.5%, 3.1% and 3.0% respectively for MOD1, MOD2 and MOD3. These values are in line with the average SEP reported for moisture meters based on NIR and Radio Frequency technologies, and are even similar to the SEP of the oven-

dry method operated with 100g samples [46]. Other non-portable technologies for MC estimation recently tested achieved lower SEP values either deploying microwave reflection operated in-line in laboratory conditions [20] or with magnetic resonance coupled with traditional sampling in ~~the~~ industrial environment [48]. Yet, ~~, but~~ being fixed equipment those provide a lower sampling flexibility.

Without assuming any particular distribution of the bias values, and with a confidence level of 90%, the statistical tolerance limits analysis reports that at least 91.5% of the distribution lies between limits with a span of 11.88, 11.13 and 10.54 percentage points respectively for MOD1, MOD2 and MOD3 (figure ~~4~~5). This result further confirms the higher accuracy of MOD3, which also features a mean value of 0.05 against 0.32 and 1.2 respectively for MOD1 and MOD2.





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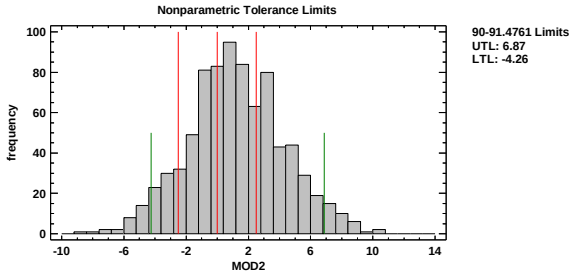

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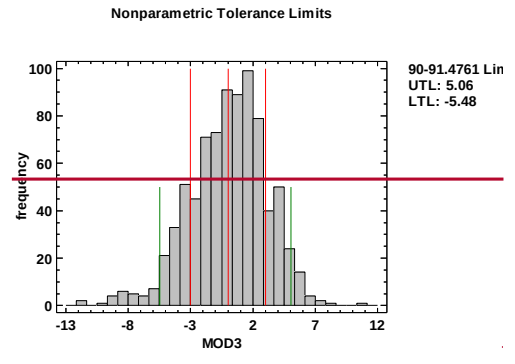

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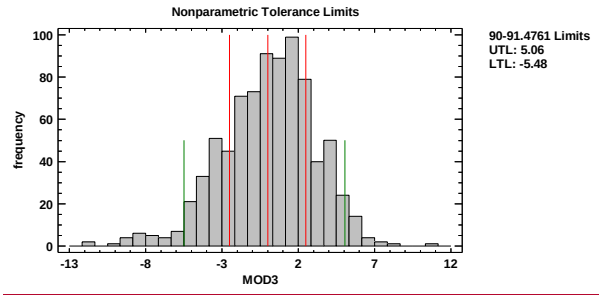

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Figure 54: Tolerance plot for nonparametric statistical tolerance limits. Green lines report the limits where 91.5% of observation lie. Red ~~dashed~~ lines represent the desired value (0, central line) and the threshold set (±2.5 points, left and right lines)

3.3 Precision

The percent of total variation due to R&R is 24.87% (table 4). Although a threshold of 10% is generally recommended (in automotive industry measurements), in other conditions with higher expected variability, values within 30% are still considered acceptable. This is surely true for MC estimation of biomass where a plethora of uncontrollable factors contribute to reducing the degree of both reproducibility and repeatability of a measurement. The value achieved is comparable to what Aminti et al. [48] reported while assessing the influence of calibration on the repeatability of a magnetic resonance MC analyzer.

Table 4: Gage r~~R~~epeatability and r~~R~~eproducibility r~~R~~eport

Measurement	Estimated	Percent	Estimated	Percent	Percent
Unit	Sigma	Total Variation	Variance	Contribution	of R&R
Repeatability	2.98922	23.8036	8.93545	5.66612	**91.63**
Reproducibility	0.903729	7.19653	0.816726	0.5179	**8.37**
R & R	**3.12285**	**24.8677**	9.75218	6.18402	100.00
Parts	12.1634	96.8586	147.947	93.816	
Total Variation	**12.5579**	100.0	157.7		

In the frame of this study, the R&R analysis highlights the differences among the prediction models in terms of repeatability, with MOD2 appearing to be the better performing in terms of deviation from average (figure ~~5~~6). Yet, just 8.37% of the total variance is due to the differences among the prediction models, being the remaining 91.63% related to the instrument. This result was partially expected, as the specific layout of the sensor deployed is designed for material more homogeneous than ~~woodchips~~wood chips. In fact, wood surface roughness is a critical factor influencing the quality and consistency of the NIR spectra even if acquired on solid timber and polished wood samples [49,50]. Thus, this aspect is magnified when measuring loose, coarse material as industrial ~~woodchips~~wood chips, leading to a less predictable illumination and reflection geometry which reduces the overall precision [49].

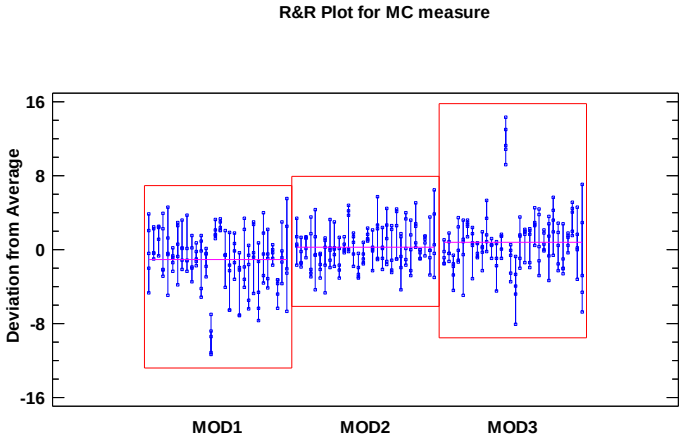

Figure ~~5~~6: R&R plot for d~~D~~eviation values. Points represents a single measurement and are grouped by prediction model (~~A~~appraiser). Horizontal red lines show the average measurement for each calibration. Vertical lines connect measurements made on the same item: the first line in each box represents the values recorded on sample 1, the second line for sample 2 and so on.

3.~~4~~3 Performance

MC measurement with the three prediction models run ~~together~~ simultaneously (thus requiring more elaboration time) took an average of 3 seconds per spectra. A whole sample, assessed with 10 replications, could be measure in about 30 seconds. In the case of laboratory analysis, sample preparation required an additional minute to arrange the ~~woodchips~~wood chips on the tray, note the ID of the sample and remove the biomass or place the tray in the oven. Overall, less than 2 minutes were sufficient to measure MC of a single sample, an analysis time comparable to that of magnetic resonance sensor [48]. Additionally, the portability of the instrument allows the operators to measure the biomass directly at the source (e.g. in the yard or on the transport unit), avoiding ~~sampling time~~the collection and transfer of samples to the laboratory and minimizing the risk of sampling and laboratory errors. Finally, the real-time response of the portable sensor permits the adoption of an adaptive measurement approach [31] when deployed in situ (e.g. ~~-~~on piled biomass or directly on the transportation unit before unloading). A high number of sampling points appropriately selected strongly enhance ~~[31], increasing~~ the overall precision of MC estimate, making it a competitive option with the current oven-drying method, which features a much longer response time and requires a higher energy input for transportation and drying of samples.

4. Conclusions

The study demonstrated the reliability of the portable NIR sensor for the determination of MC of industrial woody fuel. Among the tested prediction models, MOD3 provides the higher level of accuracy and precision. Yet, the validity of the estimate is lower when dealing with very dry (< 15%) or very wet (> 50%) samples. This drawback is probably due to the dataset used to build the prediction models: being industrial fuel, the majority of samples belonged to the average moisture classes, reducing the power of model-training in the underrepresented extreme classes. While new models based on datasets with more homogeneous distribution of MC should be developed, the tested prediction models could be still valuable in practical application. In fact, considering the different performance of the three models at the extreme values, a higher validity could be achieved by using the portable NIR spectrometer running two prediction models: MOD3 should be used as the main reference, but when both models return values above 50%, the result of MOD2 should be used, since this model features a higher validity at high moisture levels. ~~Further chemometric analysis could be developed in order to avoid the necessary operator choice. A correction factor could be considered to verify the potential improvement of models in extreme MC classes. Thus, a test has been performed correcting with different factors the MOD2 predicted MC results. A little better correlation (from 0.88 to 0.89) has been achieved, but stronger corrections could be required. The non-linearity models and the low improvement could be related to the lack of adequate number of samples corresponding to extreme values and the samples integration could enhance better performances.~~

~~Nevertheless, t~~T~~T~~he spectra acquisition is very fast, requiring about 3 seconds to return the moisture value. This performance is particularly relevant for in-field MC analysis, where the operator could gather a large quantity of spectra in a short time, reducing sampling costs and potentially applying adaptive sampling for a better estimate of the bulk quality. Future research should focus on t~~T~~his latter aspect, ~~should be object of the future research,~~ addressing the most appropriate sampling protocol for ~~moisture content~~MC determination on stock piles and in transport units. Additionally, the influence of the type of raw material (e.g. tree species or

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tree parts) and the variability due to different operators should be further investigated in order to fully disclose the potential of this technology in the bioenergy sector.

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Declaration of interests

☒The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Credit Author Statement

Giuseppe Toscano: Conceptualization, Methodology, Supervision. **Elena Leoni**: Investigation, Data curation, Formal analysis, Writing – original draft preparation. **Thomas Gasperini**: Investigation. **Gianni Picchi**: Formal analysis, Writing – original draft preparation, reviewing and editing