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

(Article begins on next page)

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Moisture content estimate of industrial wood chips by means of portable NIR spectrometer

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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 flexible tool for moisture measurement, a portable 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 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 on the fuel stocks and at different steps of the supply chain discloses a wide range of options to efficiently control fuel quality.
Suggested Reviewers:	Ana Garrido Varo, Dr- Professor, 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 asses quality parameters in a wide range of organic products
	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.
	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.
	Johanna Routa, Dr. Researcher, Luke Suonenjoki: Luonnonvarakeskus Suonenjoki johanna.routa@luke.fi Lead researcher in LUKE, member of the forest biomass laboratory is specialized in biomass quality assessment and enhancement.
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Consiglio Nazionale delle Ricerche
Istituto per la BioEconomia

Florence, December 23rd, 2021

Subject: Submission to Fuel Processing Technology

Dear Editor!

The manuscript I am submitting to FUPROC - also on behalf of my co-authors - explores the potential of a portable NIR spectrophotometer for rapid and non-destructive assessment of moisture content. The hand-held sensor is a commercial device, already used in food and pharmaceutical applications. The novelty of the research lies on the dedicated prediction models, specifically developed for solid biomass characterization.

Differently than most of the technologies for moisture content estimation, the tested NIR spectrophotometer is portable, and can be deployed directly on the fuel yard or transportation unit. This creates a wide range of possible innovations in quality control, ranging from the sampling procedure to the cost of analysis. Additionally, the paper also reports the real performance of the analyzer in term of measurement time, a crucial figure when the technology is applied in commercial applications.

To our knowledge this is the first study based on a large dataset of "commercial" biofuel assessing both reliability and "productivity" of a portable moisture meter. This topic should be relevant to the FUPROC readers, as the results provide insights on how solve one of the main challenges in bioenergy systems based on solid biofuel: the fast and reliable assessment of moisture content.

This research is in synergy with previous FUPROC papers exploring the quality assessment and management of biomass, such as:

- Gary D. Gillespie et al. (2016) The use of near infrared hyperspectral imaging for the prediction of processing parameters associated with the pelleting of biomass feedstocks. FPT 152, pp. 343-349
- Ashman J. M. (2018) Some characteristics of the self-heating of the large scale storage of biomass. FUPROC 174, pp. 1-8
- Aminti G. et al (2018) Industrial stress-test of a magnetic resonance moisture meter for woody biomass in southern European conditions. FUPROC 178, pp. 189-196

We conducted our research with the utmost scientific rigor, while trying to keep the paper as concise and easy to read as possible. We also confirm that this paper is the original product of our research. I hope that you (as well as the reviewers and the readers) will find it interesting and useful.

In the annex (following page) you can find the suggested reviewers along with the relative paper listed in the reference section.

We are looking forward to your kind feedback.

Best regards

Gianni Picchi

SUGGESTED REVIEWERS

1. Prof. Mauricio Acuna

   Reason: 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.

   Article [7] M. Strandgard, M. Acuna, P. Turner, L. Mirowski, Use of modelling to compare the impact of roadside drying of Pinus radiata D.Don logs and logging residues on delivered costs using high capacity trucks in Australia, Biomass and Bioenergy. 147 (2021) 106000.

2. Dr. Daniel Kuptz

   Reason: 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.

   Article [9] D. Kuptz, S. Lesche, T. Mendel, R. Mack, E. Rist, C. Schön, H. Hartmann, Fuel properties, dry matter losses and combustion behavior of wood chips stored at aerobic and anaerobic conditions, Biomass and Bioenergy..

3. Dr. Johanna Routa

   Reason: Lead researcher in LUKE, member of the forest biomass laboratory is specialized in biomass quality assessment and enhancement.

   Article [10] E. Anerud, D. Bergström, J. Routa, L. Eliasson, Fuel quality and dry matter losses of stored wood chips - Influence of cover material, Biomass and Bioenergy. 150 (2021).

4. Dr. Lars Eliasson

   Reason: Author of several researches analyzing the performance of different technologies for the estimation of moisture content of woody biomass

   Article [16] E. Anerud, G. Larsson, L. Eliasson, Storage of wood chips: Effect of chip size on storage properties, Croat. J. For. Eng. 41 (2020) 277–286.

5. Prof. Ana Garrido Varo

   Reason: former president of the International Council for NIR Spectroscopy is a forefront academic in the application of NIR technology to asses quality parameters in a wide range of organic products

   Article [24] Y. Pu, D. Pérez-marín, N.O. Shea, A. Garrido-varo, Recent Advances in Portable and Handheld NIR Spectrometers and Applications in Milk , Cheese and Dairy Powders, Foods. 10 (2021) 1–23.

HIGHLIGHTS

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

Moisture content estimate of industrial wood chips by means of portable NIR spectrometer

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Keywords:

NIR, moisture content, fuel quality, woodchips, 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 flexible tool for moisture measurement, a portable 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 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 on the fuel stocks 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	Repeatability 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 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, storage in uncovered yards may modify, where the biomass is exposed to uncontrollable factors such as rain, snow and wind generally leads MC increase [14,15], but it may also reduce it if the conditions are favourable [16]. This represents a further challenge as the combustion of biomass with unknown and variable MC leads to unstable and inefficient firing process [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 [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 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 directly in the field with portable instruments [23,24]. 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 [25] and the type of woody biomass [26]. 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 [27]. 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 [28,29]. 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 [30–33], leading to an additional effort to control the quality of biomass feedstock.

Finally, the availability of a portable NIR tool for the determination of MC (and other quality parameters) of woody biomass 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 [34]. 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. Yet, such development requires adequate hardware solutions and reliable prediction models to convert the raw spectra in MC figures.

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 according to the guidelines suggested by Vardeman and Jobe [35]:

- "Validity" is intended as the capacity to provide data that represent the quantity measured reliably, without the influence of factors other than the desired ones. In this case, due to the lack of information regarding the biomass quality, the unique factor considered was the influence of the extreme MC values;

- "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

<u>2.1 Woodchip samples collection and preparation</u>

The woodchip samples (n = 817) had been provided by a power plant located in Northern Italy from July to October 2020. The facility 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). All the woodchip samples had been 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. The samples had been prepared for the evaluation of MC according to the technical standard ISO 18134-1:2015.

<u>2.2 Near-infrared data acquisition</u>

NIR analysis was performed in laboratory by means of a portable MicroNIR™ OnSite sensor, which featuring no moving parts can be regarded as a “rugged” spectrometer (IP67). 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 and 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. 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 a tray where the NIR raw data was acquired on a matrix of 9 predefined spots plus a randomly-selected position as depicted in figure 1. The operation was performed manually by a unique operator throughout the whole study. After spectra acquisition the sample was oven-dried for MC measurement according to standard ISO 18134-1:2015.

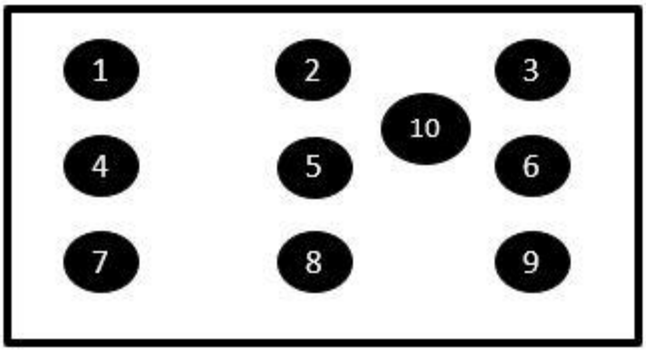


Figure 1: 3x3 matrix used for sampling NIR scans on the woodchip tray

2.3 Test of precision

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 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. These had been previously developed using the spectra acquired on different sets of industrial woodchip samples provided to the laboratory by several 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. 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 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 PLS regression 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%. The second (MOD2) and the third (MOD3) prediction models have been developed on the spectra of 212 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%. MOD3 was developed as the previous one, with additional SNV pretreatments resulting in R^2=0.94 and RMSEP=2.44%. The RMSEP values of the three models are in line or superior to the results of other researches estimating MC with NIR spectroscopy in woody materials [36,37] and other biomasses [38,39].

2.5 Data analysis

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

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

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*.

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 [40], 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 R&R test, ANOVA method. This technique is specifically designed for verifying the "Repeatibility" 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) [35]. 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.

2.5.3 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

SD: Standard Deviation; MIN: Minimum value; MAX: Maximum value.

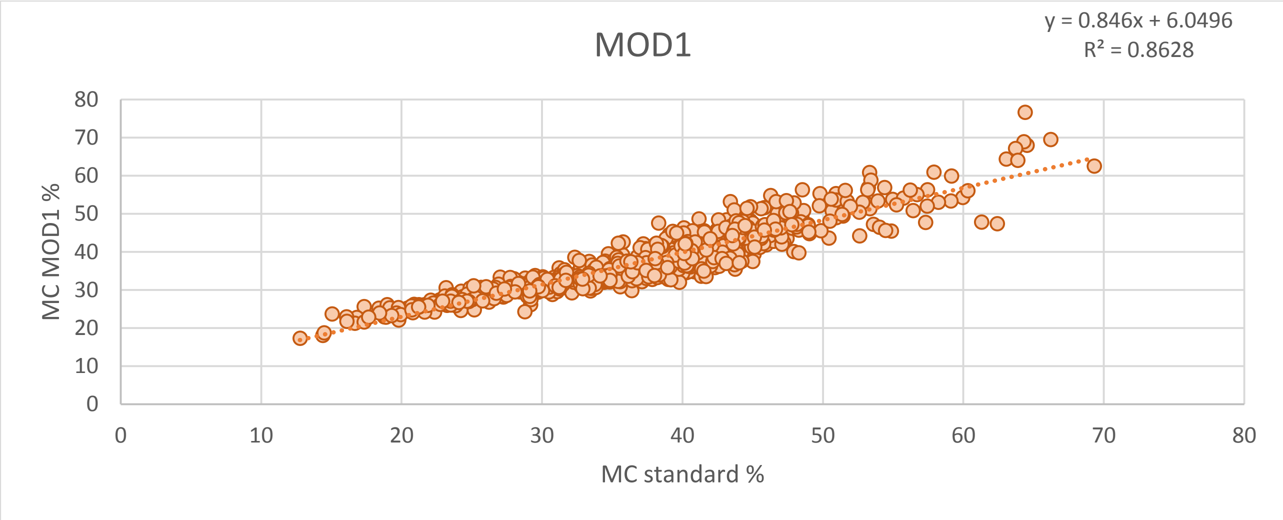
The percentage of overestimated and underestimated MC records are reported in table 2 for each prediction models. Considering absolute values, the average bias is around 2.5% for all models, while the maximum bias is produced by MOD1 (14.96%). All models show a higher frequency of overestimating occurrences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

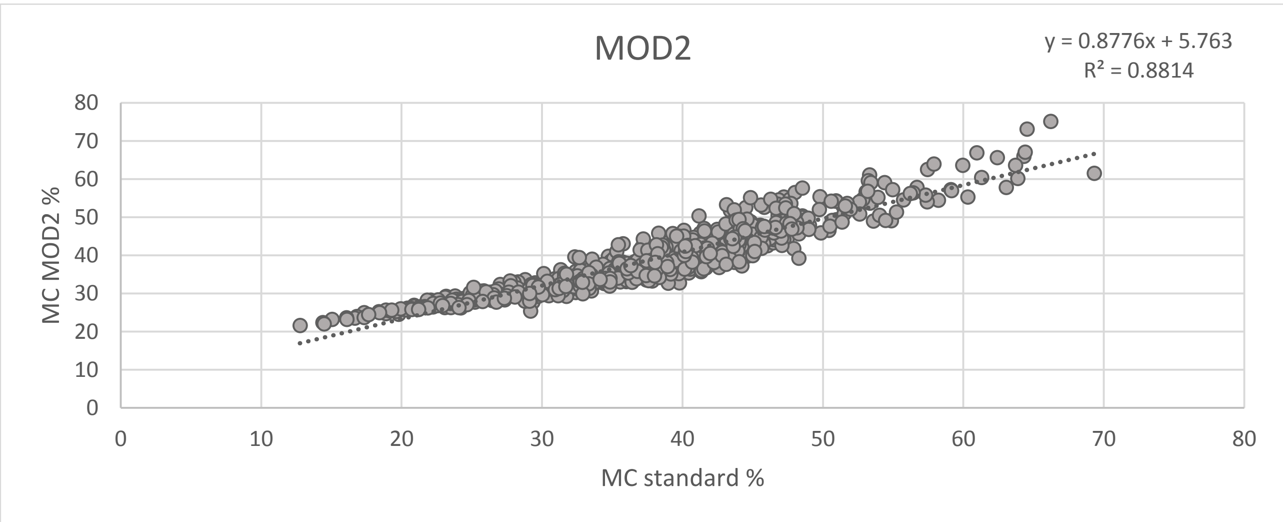
Table 2: Resulted values of bias related with each prediction model and their estimation trend

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 2). Yet, this performance is inferior to that achieved with a NIR sensor on homogeneous pelletized biomass [41] and even to that of a magnetic resonance sensor tested with samples featuring a similar variability to the present study [42]. The bias in linearity confirms the presence of some disturbance in MC estimation, with MOD3 showing the minimum deviation from linearity (β = 0.9181) to a maximum in MOD1 (β = 0.8460).





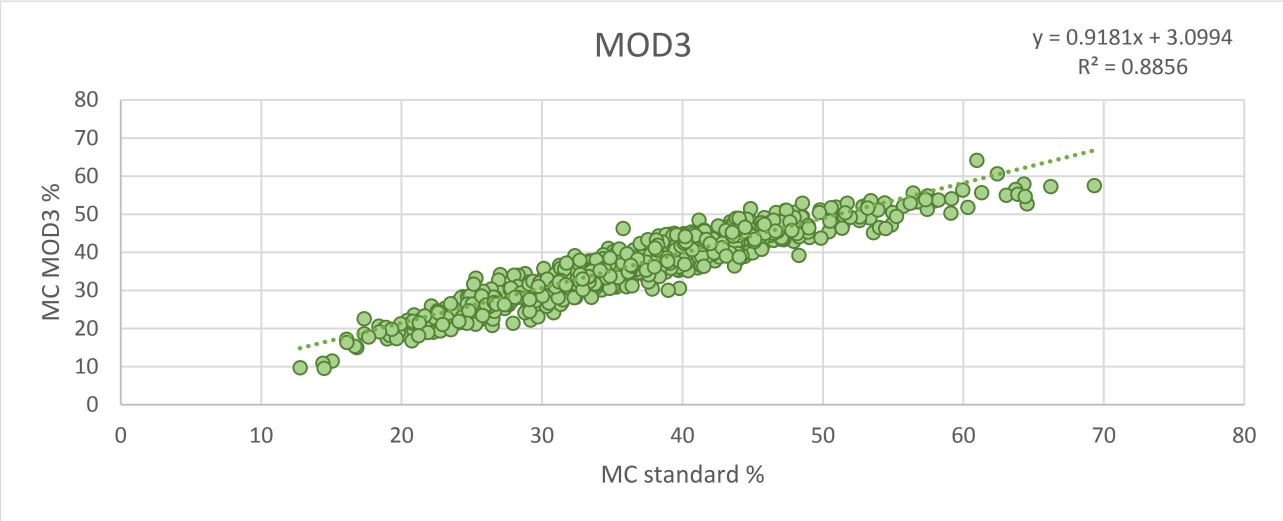


Figure 2: Relation of MC values estimated by the NIR sensor for MOD1, MOD2 and MOD3 as compared to MC estimated with standard method.

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).

MOD1 has satisfactory reliability just for the two central MC classes, with an average bias below 0.5%, while strongly overestimates drier classes and 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 the above, MOD3 appears to provide the best performance, even if its capacity to predict MC of biomass is limited to woodchips with MC lower than 50%. Above this threshold, the analysis would return a result strongly underestimated. A practical solution to this issue would be to deploy 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.

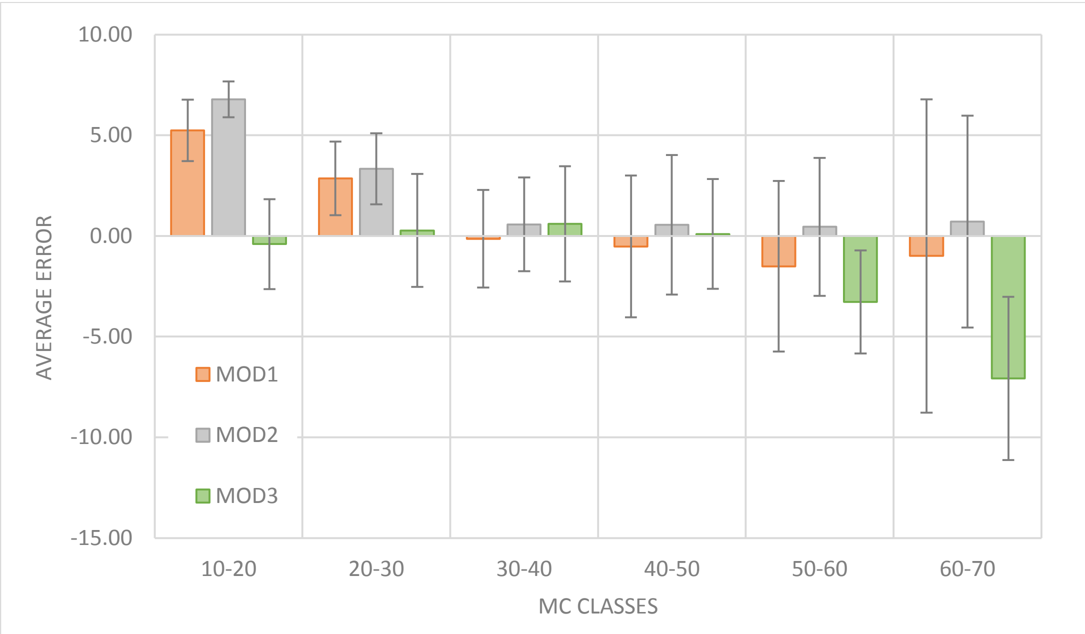


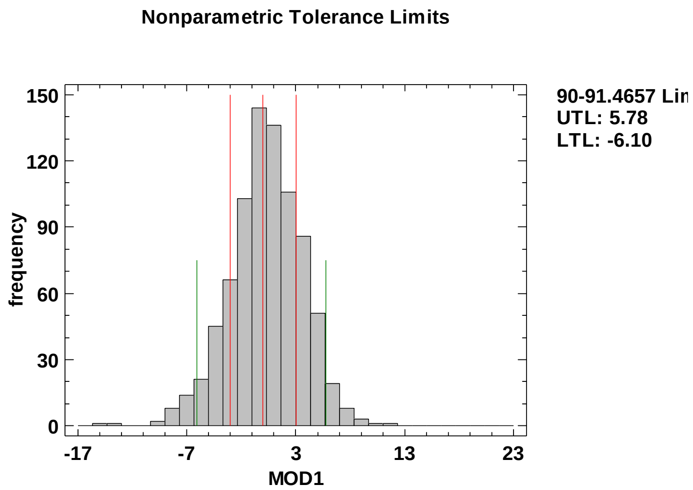
Figure 3: average error of estimate according to MC classes (as measured with standard method). Vertical bars represent the standard deviation.

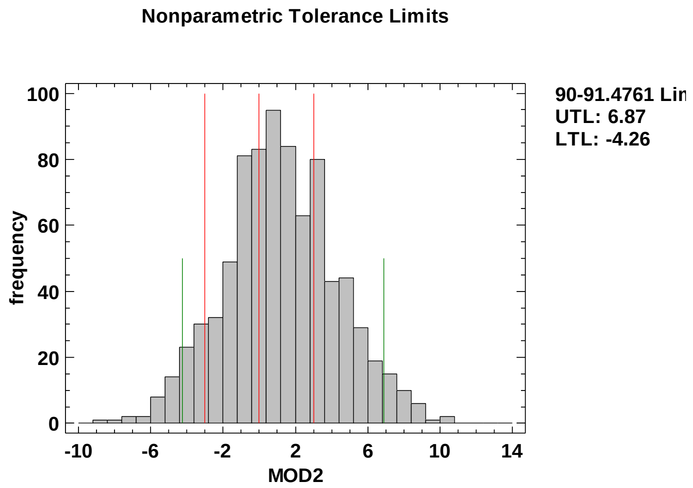
3.2 Accuracy

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 [40]. Other non-portable technologies for MC estimation recently tested achieved lower SEP values either operated in-line in laboratory conditions [20] or with traditional sampling in the industrial environment [42], but being fixed equipment 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). 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.





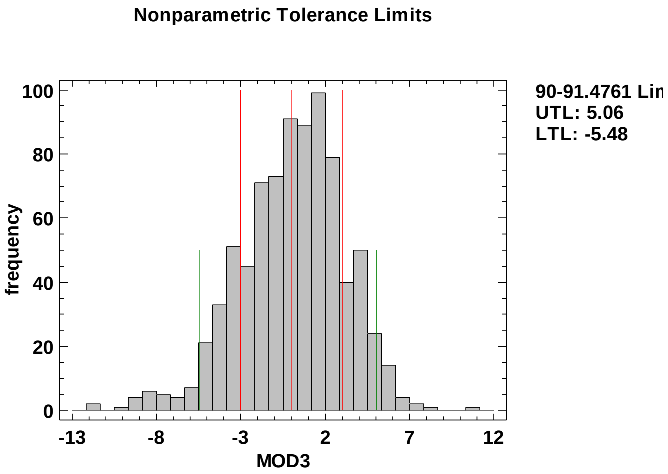


Figure 4: Tolerance plot for nonparametric statistical tolerance limits. Green lines report the limits where 91.5% of observation lie. Red dashed lines represent the desired value (0, central line) and the threshold set (±2.5 points, left and right lines)

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. [42] reported while assessing the 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 5). 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. 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 [43,44]. Thus, this aspect is magnified when measuring loose, coarse material as industrial woodchips, leading to a less predictable illumination and reflection geometry which reduces the overall precision [43].

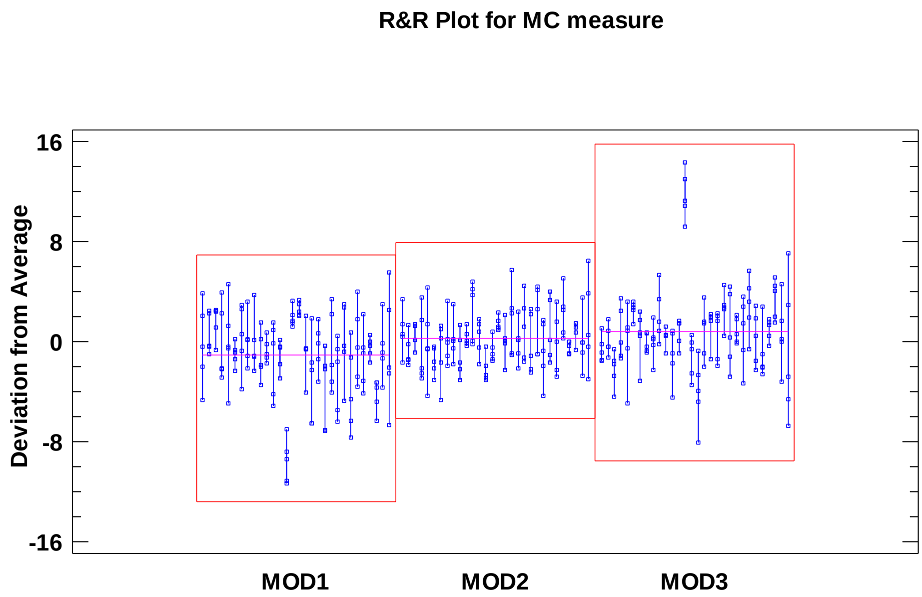


Figure 5: R&R plot for Deviation values. Points represents a single measurement and are grouped by prediction model (Appraiser). Horizontal red lines show the average measurement for each calibration. Vertical lines connect measurements made on the same item: the first line in each box represents the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

3.3 Performance

MC measurement with the three prediction models run together (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 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 [42]. 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 and minimizing the risk of sampling errors. Finally, the real-time response of the portable sensor permits the adoption of an adaptive measurement approach [27], increasing the overall precision of MC estimate.

4. CONCLUSION

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 or very wet 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. This latter aspect should be object of the future research, addressing the most appropriate sampling protocol for moisture content determination on stock piles and in transport units.

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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: