



Comparison of the performance of a microwave-based and an NMR-based biomaterial moisture measurement instrument

Petri Österberg^{1,2}, Martti Heinonen³, Maija Ojanen-Saloranta³, Anssi Mäkynen¹

¹University of Oulu / CEMIS-Oulu, Kehräöntie 7, FI-87400 Kajaani, Finland

²Kajaani University of Applied Sciences, Kuntokatu 5,, FI-87101, Kajaani, Finland

³VTT Technical Research Centre of Finland Ltd, Centre for Metrology MIKES, Tekniikantie 1, 02150 Espoo, Finland

ABSTRACT

This article compares the performance of an NMR-based and a microwave-based moisture measurement instrument designed for biomaterials. The conventional moisture measurement method, Loss-on-Drying, serves as a reference measurement for both instruments. Six different biomaterials at three moisture content levels were measured with the microwave instrument and five biomaterials were measured with the NMR instrument. After instrument calibrations, the difference and variation of the measurement results for parallel samples and the repeatability of measurement results obtained by the NMR and microwave instruments were estimated. Reasonable agreement between the measurement methods was achieved.

Section: RESEARCH PAPER

Keywords: moisture measurement; bioenergy; microwave; NMR; Loss-on-Drying

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Corresponding author: Petri Österberg, e-mail: petri.osterberg@measurepolis.fi

1. INTRODUCTION

The Loss-on-Drying (LoD) method is conventional and is still the only standardized method of measuring the moisture content of biomaterial load. To determine the moisture content of the load, a sample of at least 300 g is taken from the biomaterial load of a truck or trailer according to an appropriate sampling standard [1]. The sample is milled to reduce the maximum particle size to meet the requirements of a sample preparation standard [2]. Finally, the moisture content of the biomaterial sample is determined according to the standardized LoD method [3], that is, by weighing the sample and then drying it for several hours in an oven at a temperature of 105 ± 2 °C and performing final weighing after the moisture of the sample has been fully removed (i.e. after the mass change of the sample is below the detection limit given in the standard). However, the drying time should not exceed 24 hours. After

the final weighing, the moisture content of the sample is calculated as the ratio of the mass change to the mass of the original sample. The LoD procedure is too slow for efficient process control, but if not in all countries, at least in several countries it is still the only standardized moisture measurement method for the moisture-content determination of biomaterial delivery lots and is therefore used mostly in invoicing. In a typical case at a power plant, the LoD-based moisture content estimate for the biomaterial load is available from two days after delivery.

To overcome the problem of the slowness of the LoD method, novel rapid moisture measurement methods have challenged the conventional oven drying method. In this research, we studied two of these new methods, namely the nuclear magnetic resonance (NMR)-based moisture-measurement solution developed by Valmet Ltd, called the MR

Moisture Analyzer, and the microwave-based moisture measurement solution developed by Senfit Ltd, called the BMA Desktop. The calibration of the NMR instrument is very simple: it is done with a water container, first empty and then filled, whereas the microwave instrument calibration is more complicated: it must be calibrated separately for each biomaterial section with the reference LoD method. However, the microwave instrument is more rapid than the NMR instrument: a single measurement takes only ten seconds, whereas an NMR measurement takes two minutes. Still both are very rapid in comparison with the conventional method: the LoD-based moisture content estimate takes at least 16 hours. However, new research results are urgently needed to demonstrate the performance of novel instruments designed for delivering moisture data for biomaterial invoicing.

This article is constructed using the research and data published and presented at two scientific conferences [4], [5]. The article describes a comparative study of two novel alternative instruments designed to mostly or fully replace the use of the conventional oven drying method. The benefits and drawbacks of the NMR and microwave instruments and their suitability for power plants are compared and discussed.

During our research, Swedish and Canadian researchers published an article [6] in which they described the testing of the Metso (now renamed as Valmet) MR Moisture instrument with forest-based biomaterial samples with different moisture contents in both countries. In comparison with our measurements, the measurement difference from the oven drying was smaller in Fridh and Eliasson's results in Sweden but larger in Volpé's results in Canada, and the repeatability of the measurements was also worse in the research results obtained in both countries. In the article [6], the researchers used four to five moisture classes per material, whereas in this article three moisture classes but more sample materials and more samples per material and per moisture class were measured. One of the Swedish authors of the previous publication, Fridh, also published a working report on the NMR instrument for Skogforsk at the same time [7]. In comparison with reference oven drying, the difference and standard deviation given by Fridh's results were slightly smaller than in this article. In addition to these, some publications of the moisture measurement instrument prototypes using NMR technology are available [8], [9].

Only a few publications about commercial biomaterial moisture measurement instruments based on microwave technology could be found during the research. Two theses written in Finnish described the performance of corresponding Senfit BMA Desktop microwave instruments [10], [11], but they researched the performance with selected biomaterials in a restricted moisture range, namely within the range of moisture contents at delivery.

The research described in this article is part of a European project, METefnet [12], which concentrates on the creation of unambiguous SI (Système international d'unités i.e. the International System of Units) traceability chains for measurements of moisture in solid materials.

Section 2 describes the solid biofuel sample materials, sampling, and sample preparation procedures used during the research. In Section 3 we will present the research instruments. Instrument calibrations are described in Section 4. Section 5 describes how the moisture measurement process with all the three measurement methods – the NMR-instrument, the microwave instrument, and the reference LoD method – is

performed. In Sections 6 and 7, measurement results are presented for the NMR instrument and the microwave instrument respectively. The discussion in Section 8 compares the performance of the measurement instruments. Finally, we conclude the article and the future plans are discussed.

2. BIOMATERIAL SAMPLES AND SAMPLE PREPARATION

The biomaterial samples for the research were collected from two locations: from a nearby sawmill and from lorries transporting solid biofuel loads to a local power plant. Six different biofuel sections presented in Figure 1 were chosen:

1. Sawdust from a sawmill
2. Bark waste from a sawmill
3. Chipped pruning residues
4. Chipped small-sized trees (mixed with sawdust)
5. Crushed and chipped stumps
6. Milled peat

However, milled peat samples were not measured with the NMR instrument but only with the microwave instrument. This was due to the possible ferromagnetic components in peat and is discussed more detailed at the end of Section 3: 'Research Instruments'.

Two measurement sessions were arranged, but the microwave instrument was available only for the first session. The NMR instrument was available for both sessions. For the first measurement session in October 2014, bark waste and pruning residue samples were stored for several months in a pile before measurement so that the original greenish colour of bark samples changed fully to grey/brown and the green needles in pruning residue samples dried and dropped away. For the second measurement session in May 2015, bark waste samples and pruning residue samples were fresh and collected only some hours after debarking of the logs and delimiting of the trees. For both measurement sessions, sawdust samples were fresh and stumps were stored for several months in a roadside storage area before processing and measurement. Chipped small-sized trees were not delivered to the power plant during the second measurement session and this sample material could be measured only during the first measurement session.

About 50 L of each selected biofuel section was collected. Each biofuel section was taken from a single location of the same truckload or storage pile to ensure the similarity of parallel samples for the research. This was the opposite of the normal sampling procedure, in which a biofuel sample is collected from several locations of the load or pile to ensure that the sample represents the whole load or pile.

All biofuel sections were measured at three different moisture levels. Thus the 50 L section samples were divided into three parts. The first part was measured at the moisture content present at delivery. The second part was spread in a shallow empty pool for drying at room temperature for three to five days depending on the initial moisture and the drying speed of each biomaterial. The third part of the biofuel sample was placed in a water tub and additional water was inserted at the beginning to increase the moisture content of the sample. The amount of water added was determined according to the moisture content at delivery of each biofuel section. The sample was stored for two days in the water tub with a lid. After the two-day moistening period, the free water was removed from the moisturized biomaterial. Altogether, there were 15 different biofuel sample sets for the NMR instrument research and 18



Figure 1. Solid biofuel samples from upper left corner: sawdust, non-fresh bark waste, non-fresh pruning residue, chipped small-sized trees, crushed and chipped stumps, and milled peat.

sets for the microwave instrument research, that is, five biofuel sections at three moisture-content levels for the NMR instrument and six biofuel sections at three moisture-content levels for the microwave instrument.

Samples were milled to meet the particle size requirements of the sampling standard for the oven drying method [2] (the particle size must be smaller than $31\text{ mm} \times 31\text{ mm} \times 31\text{ mm}$ for oven drying). Before a measurement session, each biomaterial sample of ca. 15 kg at three moisture levels was mixed separately in a cement mixer to obtain homogenous sample material. Homogenized sample material was divided into ~ 1 kg subsamples in plastic bags. The plastic bags were closed so that as little air as possible remained in them. Before the moisture content measurement, the biomaterial subsamples were stored for one night at room temperature to obtain the constant temperature and moisture content of the biomaterial samples inside the plastic bags.

The number of samples was from 6 to 20 pieces for each biomaterial and moisture level per instrument, depending on the biomaterial and moisture content (6 to 13 samples for the microwave instrument and 8 to 20 samples for the NMR instrument). Altogether the moisture contents of 398 biomaterial samples were measured in this study with the NMR moisture measurement instrument and 191 samples with the microwave one. Corresponding LoD reference measurements were also made for the samples (589 LoD measurements). Additionally, some of the samples were measured several times to test the repeatability of the NMR and microwave instruments.

The volume of biomass samples for the NMR instrument was always 0.8 L, but the sample weight varied between 161.4 and 492.1 g, whereas the sample weight for the microwave instrument was always 400 ± 1 g, but the sample volume varied according to the material and moisture content.

3. RESEARCH INSTRUMENTS

Four ovens were used as the reference moisture measurement instruments to dry out the biomaterial samples during the measurement session. All the ovens were made by Termaks [13], and they included two TS4115 models and two newer models, TS8056 and TS8136. A single oven can simultaneously dry 6 to 18 biofuel samples of about 400 g depending on the oven model and measures of sample containers. Precisa EP 2220M and Sartorius CP4202S scales were used in the weighing of the LoD samples.

Two Valmet MR Moisture Analyzers [14] were used as the NMR-based instrument in this study (see the left part of Figure 2). In the first measurement session, an earlier version of the same instrument was used; it was called the Metso MR Moisture Analyzer prior to the company's demerger. The moisture content measurement range of the instrument is 0–90 %. The instrument measures biomaterial samples in the 0.8 L measurement container seen in the image on the right side of Figure 2. The container is inserted in the measurement chamber and the measurement of the moisture content of a sample takes two minutes. The measurement chamber is located inside a magnet, and thus samples having ferromagnetic components may cause problems during the measurement. For example, peat samples may contain ferromagnetic components and thus the instrument's manufacturer does not recommend their use as measurement samples.

Senfit BMA Desktop [15] was used as the microwave-based instrument in this study (see the left part of Figure 3). The moisture content measurement range of the instrument is 0–70 %. The instrument was tuned for a 400 ± 5 g biomaterial sample placed in a plate-shaped measurement bowl seen on the right side of Figure 3. The bowl is inserted in the measurement chamber and the moisture measurement of the sample takes



Figure 2: Valmet MR Moisture Analyzer. Left: NMR moisture measurement instrument; right: measurement container of the instrument with sawdust sample.

about ten seconds. The particle size of the sample should be no more than 31 mm × 31 mm × 31 mm; thus all the samples were milled with a Senfit Sample Mill [16] to obtain the appropriate particle size.

4. INSTRUMENT CALIBRATIONS

Before the start of the measurement session, the drying ovens were tested and calibrated against a calibrated thermometer to ensure that they met the requirements of the standard [3]. The temperature must stay between 103 and 107 °C during the drying time, that is, at least 16 hours. The maximum drying time is 24 hours according to the standard.

The calibration of the NMR moisture measurement instrument is very simple. One 0.8 L sample container was filled with water, closed with a lid, and stored for a day in a measurement room so that the water temperature equalled the temperature of the sample stored in the measurement room. Another sample container was left empty. Every morning before the actual measurements, these two containers were measured to calibrate the NMR instrument. The calibration water in the container was not changed during the measurement session. The calibration procedure took about five minutes.

Careful calibration of the microwave moisture measurement instrument is essential to achieve reliable measurement results. The calibration of the microwave instrument is done with calibration samples whose moisture content has been determined using the LoD method. The closer the properties of the calibration material are to the properties of the sample material, the better the measurement results that are achieved. The calibration of the microwave instrument should be supplier-specific for every biomaterial section and should be done prior to the biomaterial moisture measurements. Although the measurement range of the microwave instrument is a moisture content of 0 to 70 %, the instrument must be calibrated for the moisture content ranges of 0–15 % and 15–70 % separately for all material sections. However, solid biofuels drier than 15 % are very rare at combustion plants.

The measurement range in this research was planned to be 15–70 % and the microwave instrument was calibrated for this range. The calibration was carried out by using calibration samples taken from the same truck load and location as the

measurement samples. Two calibration samples were picked out from each sample material set and moisture level after milling (to meet the particle size requirements of the standard) and mixing the biomaterial. Thus the calibration curve was derived from six calibration points for each biomaterial (two samples at three moisture levels).

We also tested a calibration case that is weaker, but still possible in real life at power plants. According to the instructions given by the instrument manufacturer personnel, two samples of each material were dried or moistened to four moisture levels so that there were eight calibration points for each biomaterial section to create a calibration curve for the microwave instrument. Unlike in the previous calibration, the calibration samples were collected one to three weeks before carrying out the measurement procedure on the research samples. Thus in this case the calibration samples were taken at least from different loads, but likely from a different forest stand or roadside storage area too. For five out of the six materials, the driest calibration samples had moisture contents slightly below 15 % (the final moisture content was between 9 and 12 %) and thus the calibration curve was slightly distorted at the drier end. Additionally, two of the six biomaterial suppliers (suppliers for chipped small sized trees and milled peat) changed between the collection of the calibration samples and the measurement session. These factors probably decreased the measurement accuracy considerably, in spite of the fact that the measurement samples were visually the same as the calibration samples.

5. MOISTURE MEASUREMENTS

During the research, two moisture measurement sessions were arranged. The first measurement session was performed in autumn 2014 and the second in spring 2015. The NMR instrument was available for both sessions and the microwave instrument was available for the first measurement session. Additionally, all the samples were measured with the LoD reference method. The NMR instrument was changed for the second measurement session, but it was similar to the instrument used in the first measurement session. Before the measurements, the biomaterial samples of ca. 1 kg were stored overnight in plastic bags at room temperature to obtain a constant temperature and moisture content inside the plastic

bag. The measurement procedure of a single sample started by shaking a plastic bag to mix the sample and the bag was emptied onto a clean table. The sample was divided into two parts. The first part was a sample of $400 \text{ g} \pm 1 \text{ g}$, which was weighed and placed in the measurement bowl of the microwave instrument seen on the right side of Figure 3. The microwave moisture measurement takes about ten seconds and the instrument saves the measurement data. The instrument measures the sample temperature using an IR-sensor and tunes the microwave moisture measurement result based on the temperature of the sample. The second sample part is placed in the 0.8 L NMR instrument sample container seen on the right-hand side of Figure 2. The NMR instrument weighs the sample, measures the moisture content of the sample in two minutes, shows the result on the screen, and saves the measurement data. After the microwave or NMR moisture measurement, the biomaterial sample was placed in a metal container, weighed, and inserted in the oven for the reference moisture measurement with the LoD method. The drying time was chosen as 23–24 hours, ensuring at least 16 hours heating at $105 \text{ }^\circ\text{C}$ to meet the requirements of the standard [3]. Due to the long drying time and large number of samples, several ovens were used to achieve an efficient operation. After the drying period, the sample was weighed again. The obtained mass loss represents the vaporized moisture from the sample. Finally, the moisture content estimate for the sample was calculated by dividing the mass loss by the initial sample mass.

The repeatability of the microwave and NMR instruments' results was tested by repeating the measurement of the same sample five times. The moisture contents of 18 (with the microwave instrument) and 15 (with the NMR instrument) different biomaterial samples (six and five biomaterial sections, respectively, at three moisture levels) were measured five times each. The sample was removed and placed back in the measurement chamber of the instrument between separate measurements. The time gap between consecutive NMR measurements was at least five minutes to avoid warming of the samples inside the NMR instrument. The time gap between consecutive microwave moisture measurements was shorter, because sample warming is negligible in the microwave instrument. The operating power of the microwave moisture

measurement instrument is about 10 mW and thus very small in comparison with, for example, a microwave oven. The sample container of the microwave instrument was turned by about 30° between consecutive measurements. The position of the sample container inside the NMR instrument was random.

Altogether 923 separate measurements were made during this research. The measurements include the NMR and microwave instrument performance measurements, the repeatability measurements, and the corresponding LoD reference measurements.

6. MEASUREMENTS RESULTS OF NMR INSTRUMENT

6.1. Common NMR instrument results for all biomaterial samples

As stated in the Introduction, most of the measurement results and the data published in Section 6 were included in the conference proceedings [5]. The comparative analysis of the two instruments based on the previously published data is the additional information provided in this article. The representation of the research results has also been improved.

For five chosen biomaterials, the average difference between all the moisture measurement results determined with the NMR instrument and the oven drying method is $1.0 \pm 3.8 \text{ \%mc}$ (percentage points of moisture content) when the uncertainty is given at the 95 % confidence level ($k = 2$). Thus the NMR instrument overestimated the moisture content of the sample by about 1 %mc in comparison with oven drying. The moisture range for five different forest-based biomaterials varied from 14.3 to 68.6 %. On average, the standard deviation of moisture measurements for parallel samples was 0.9 %mc for the NMR measurement instrument and 0.4 %mc for the oven drying method, when considering all biomaterials at three moisture levels. Thus, the variation of the measurement results for parallel samples of single material is smaller with the LoD method.

The repeatability tests for the NMR instrument showed that the standard deviation for the measurement repeated five times on single samples taken from all five sample materials at three moisture levels was 0.5 %mc on average. Repeated measurement results seemed to deviate randomly. The material-



Figure 3. Left: Senfit BMA Desktop, the microwave moisture measurement instrument; Right: plate-shaped measurement bowl of the instrument with a crushed and milled stump sample divided into three parts.

specific and moisture-level-specific repeatability tests are described in Section 6.3. Obviously, the five repeated measurements on the single sample cannot be done for the oven drying method, because the sample changes (dries) during the first measurement.

6.2. Biomaterial-specific measurement results for the NMR instrument at three moisture levels

This section summarizes the biomaterial-specific and moisture-level-specific NMR moisture measurement results for all the biomaterials compared with the reference LoD measurements.

Figure 4 shows the performance of the NMR measurement instrument for sawdust, bark waste, and pruning residue samples in comparison with the LoD method by showing all the measurements with red crosses (the first NMR instrument and measurement session) and blue circles (the second NMR instrument and measurement session). The x-axis represents the difference in moisture measurements between the NMR instrument and the reference LoD method, while the y-axis represents the moisture content of the sample measured with the reference LoD method.

Sawdust is typically a quite homogenous material and the uncertainty was the second smallest for the NMR instrument after measurements of crushed and milled stump samples (NMR – LoD differences of stump samples can be seen on the left-hand side of Figure 5). Here, the mean difference in moisture content between the oven-drying method and the NMR instrument was 1.6 ± 2.1 %mc for sawdust when a 95 % confidence level was applied ($k = 2$). In this case, the measurement results made with the second instrument during the spring 2015 measurement session were very good and were closer to the reference LoD results than the measurement results achieved by the first measurement session and instrument.

The middle image of Figure 4 shows the performance results

of the NMR instrument with bark waste samples. For the first measurement session, chipped bark samples were collected from the unloading and feeding station of the combustion process of a power plant. This means that bark material was stored in the pile for some weeks before the measurement so that the greenish colour of the sample material vanished and the solid biofuel became acceptable for combustion at the power plant. For the second measurement session, bark samples were fresh and greenish and were collected some hours after debarking the logs. Despite that, the results of both measurement sessions are quite similar, as seen in the middle image of Figure 4. For chipped bark samples, the deviation of the NMR instrument results from the reference LoD method results was 3.0 ± 2.4 %mc on average when a confidence level of 95 % was applied. Thus bark waste was the biofuel whose moisture content was most overestimated by the NMR instrument.

The right-hand image in Figure 4 shows the performance results of the NMR instrument with pruning residue samples. For the pruning residue samples, the mean deviation between all the NMR moisture measurement readings and the reference LoD method readings was -0.9 ± 4.1 %mc when a 95 % confidence level was applied. Thus the pruning residue was the only biofuel whose moisture content was underestimated by the NMR instrument. However, when observing the right-hand image of Figure 4, it seems that the NMR instrument underestimates the moisture content mostly with dry samples. Pruning residue is very inhomogeneous material and that may be the reason why the standard deviation and the uncertainty, respectively, were quite large and nearly doubled in comparison with the other four measured forest-based biomasses. In particular, the deviation in moisture content of the moistened pruning residue samples measured in the first measurement session was large on the right side of Figure 4. The largest single difference between the NMR instrument and the reference LoD method measurement results is no less than ~ 6

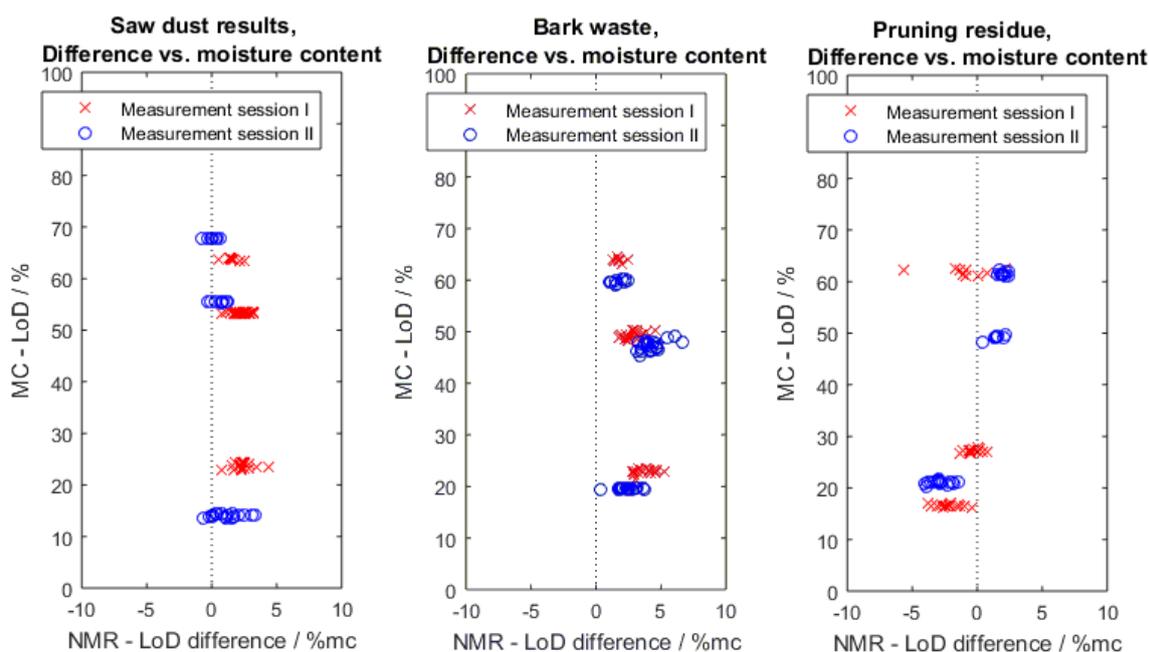


Figure 4. The differences of all measurement pairs between the NMR measurement and the reference LoD method are presented for sawdust (left-hand image), bark waste (middle image), and pruning residue (right-hand image). The x-axis represents the NMR – LoD difference in moisture content while the y-axis represents the moisture content of the sample according to the reference measurement. If the circle or cross falls on the dashed line, no deviation between the NMR and reference instrument measurements exists. The corresponding images of the remaining biomaterials are presented in Figure 5.

%mc in the right-hand image of Figure 4. Typically, the reason for such outliers was incorrect weighing (either manual weighing during the LoD method or automatic NMR weighing), but the reason for this outlier could not be found and therefore this is included in the analysis. For the first measurement session, the pruning residue sample material was collected from a truckload at the unloading station of the power plant, and before that the samples were stored for several months in a pile in a forest roadside storage area to get rid of the needles in order to increase the fuel energy content and decrease the harmful corrosive components of the solid biofuel. Most needles dried and dropped away during the storage period and transportation. For the second measurement session, the sample material was collected from the forest stand during logging, and thus the fresh and green pruning residue sample material with needles was used in this measurement session. However, a considerable difference in measurement results was not obtained. In both cases the moisture content was underestimated with dry samples and overestimated with wet samples in comparison with the LoD measurement and this explains the higher standard deviation. The freshness of the pruning residue samples of the first and the second measurement sessions can also be noticed in the right-hand image in Figure 4: the moisture content at delivery is about 27 % in the first measurement session (i.e. the most middle cluster of the three clusters of measurement results marked with red crosses) while the moisture content at delivery in the second measurement session is about 50 % (i.e. the middle-most cluster of the three clusters of measurement results marked with blue circles).

The right-hand image in Figure 5 shows the deviation between the NMR instrument and the reference LoD method measurement pairs for chipped small-sized trees. The sample material of chipped small-sized trees was available only for the first measurement session and the deviation of the NMR measurement results in comparison with the reference LoD

method was 0.7 ± 2.6 %mc on average when a 95 % confidence level was applied. During the collection of chipped small-sized tree material samples, we noticed that the chosen biomaterial was not pure but was mixed with sawdust, which may affect the results in comparison with pure material. The measurement results here, indeed, were quite close to the sawdust results, although the material seemed totally different, when observing the left-hand images in the upper and lower corners of Figure 1.

The crushed and chipped stump material contained more soil than the other four solid biofuel sample materials, and thus weaker NMR measurement results were expected. However, both differences in comparison with the reference LoD method and the $2 \times$ sigma based measurement error were the smallest among the five tested biomaterials, being 0.1 ± 1.8 %mc. The right-hand image in Figure 5 shows that the measurement differences from the reference LoD method results and their variation are small and the results here are beautifully concentrated quite close to the zero line.

Biomaterials are typically very inhomogeneous and the variation of moisture measurement readings between parallel samples was notable even for sawdust. The standard deviation of moisture readings of parallel samples obtained by the oven drying method varied from 0.0 %mc to the poorest case of 1.4 %mc depending on the sample material and moisture level. The mean of these standard deviations was 0.4 %mc. Correspondingly, the standard deviation of moisture readings of parallel samples obtained by the NMR moisture measurement instrument varied from 0.4 %mc to the poorest case of 2.1 %mc depending on the sample material and the moisture level. The mean of the standard deviations of NMR measurements was 0.9 %mc. A clear dependence between the moisture content of the solid biomaterials and the standard deviation of the parallel measurements could not be observed, even though the dependence between the moisture content of the biomaterial and NMR instrument repeatability for the very

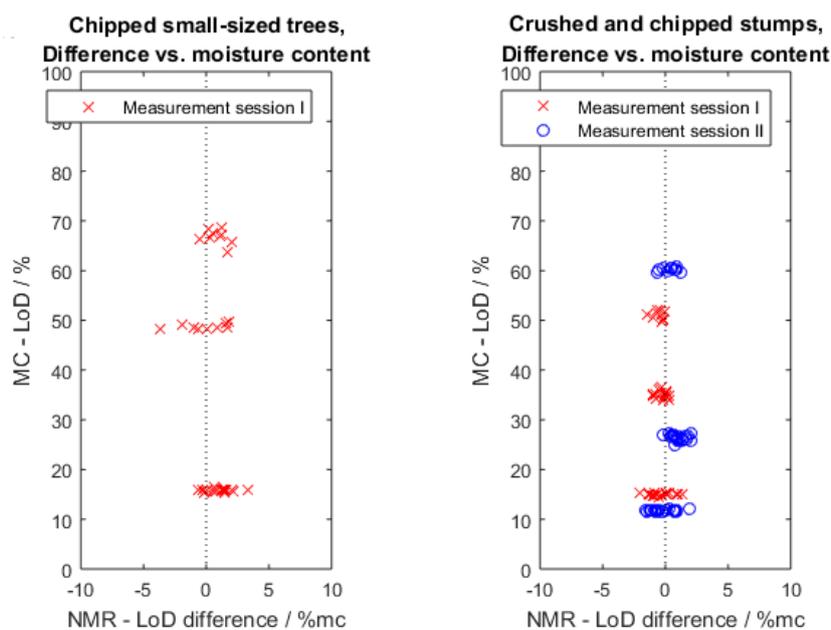


Figure 5. The differences of all measurement pairs between the NMR measurement and the reference LoD method are presented for chipped small-sized trees in the left-hand image and for crushed and chipped stumps in the right-hand image. The x-axis represents the NMR – LoD difference in moisture content and the y-axis represents the moisture content of the sample according to the reference measurement. If the circle or cross falls on the dashed line, no deviation between the NMR and the reference instrument measurements exists.

same sample is obvious, as described later in Section 6.3.

Four of the 388 measurement pairs were omitted from the analyses due to incorrect sample weighing by the NMR instrument. The scale of the NMR instrument showed the masses of those four samples to be tens of grams different from the real masses. In addition to weighing by the NMR instrument, the sample must be weighed manually before oven drying, and the result must be quite close to the NMR weighing result. Also, the masses of parallel samples should be rather close to each other, because the sample container must always be completely full. If considerable deviation between parallel samples or between NMR and manual weighing is observed, it can be supposed that there is a weighing error. Two of 388 measurement pairs were omitted due to incorrect handling of samples during LoD measurements.

The milled peat sample material was also available during the measurements, but it was measured only with the microwave instrument. The NMR instrument manufacturer does not recommend the use of peat samples due to the fact that the measurement is based on a homogenous magnetic field. Namely, some peat materials from certain geological areas may have ferromagnetic components, which may distort the magnetic field inside the NMR instrument and further distort the moisture measurement readings.

6.3. Repeatability measurements of the NMR-based moisture measurement instrument

Table 1 summarizes the material-specific and moisture-level-specific repeatability tests of the NMR instrument for single samples. One sample of each sample class was chosen, the NMR moisture measurement was repeated five times on the sample, and the average moisture and standard deviation of the five measurements were calculated. Table 1 shows that the standard deviation of the repeated measurement results varied from 0.2 to 1.1 %mc, depending on the biomaterial. The average of all standard deviations of single samples for different

Table 1. The repeatability test results of the NMR measurement instrument for the five solid biomaterials at three moisture levels.

Moisture class	Autumn 2014		Spring 2015	
	Moisture on avg. /%	Std /% mc	Moisture on avg. /%	Std /% mc
Sawdust				
- dried	26.2	0.7	14.7	1.1
- normal	54.7	0.2	54.9	0.4
- moistened	64.4	0.2	68.4	0.3
Bark waste				
- dried	26.4	0.7	21.3	0.9
- normal	50.9	0.2	50.4	0.7
- moistened	64.7	0.2	59.6	0.2
Pruning residues				
- dried	15.9	1.1	18.8	1.0
- normal	27.1	0.9	51.8	0.2
- moistened	58.1	0.4	64.0	0.2
Small-sized tree				
- dried	17.2	0.7	-	-
- normal	49.9	0.7	-	-
- moistened	64.2	0.2	-	-
Crushed stump				
- dried	15.9	0.5	11.1	0.4
- normal	35.4	0.5	27.7	0.5
- moistened	50.3	0.3	58.7	0.9

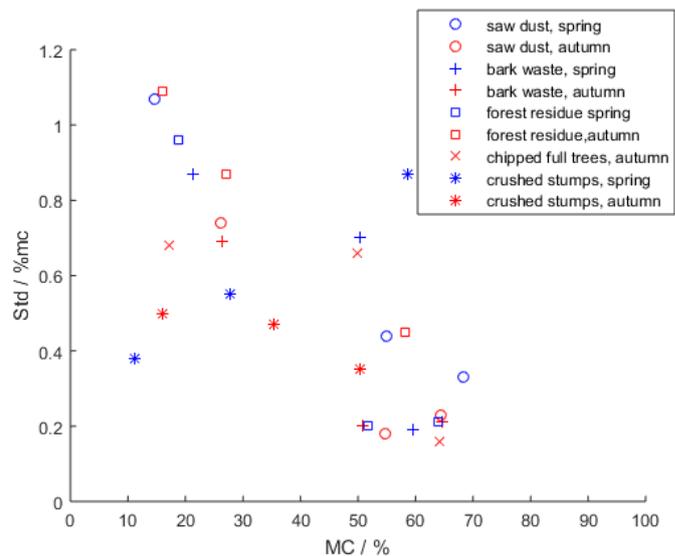


Figure 6. The graph presents the values from Table 1 graphically, and the dependence between repeatability and moisture content can be clearly seen.

materials and moisture levels was 0.5 % and the higher standard deviation values were achieved with drier samples. In Section 6.1, we showed that the standard deviation with parallel samples (not exactly the same) was 0.9 %mc on average; thus roughly half of the variation of the NMR instrument results can be explained by the variation of the biomaterial and another part can be explained by the instrument properties. The repeatability test values from Table 1 are plotted on a graph (see Figure 6, right). The graph clearly shows that the standard deviation of consecutive measurements of a single sample increases when the sample is drier. Thus repeatability is better with moister samples. However, this dependence vanishes when variation due to the material properties is added, as mentioned at the end of Section 6.2. The repeatability test results for the small-sized tree samples in the spring 2015 measurement session are missing from Table 1, because this solid biofuel material was not available during the second measurement session.

7. MEASUREMENT RESULTS OF MICROWAVE INSTRUMENT

7.1. Common microwave instrument results for all biomaterial samples

Similarly to the NMR research data and measurement results, most of the measurement results and the data obtained with the microwave instrument were included in another conference proceedings [4]. The representation of the research results of the microwave instrument has also been improved and the measurement results are discussed from the viewpoint of the differences between the instruments.

The measurements made with the microwave-based moisture measurement instrument were similar to those made with the NMR moisture measurement instrument in Section 6. However, the samples were measured less, because the microwave instrument was available only for the first measurement session. Contrary to the NMR measurements, the peat samples were measured with the microwave instrument.

For six chosen biomaterials, the average difference between all the moisture measurement results determined with the microwave instrument and the oven drying method is 0.0 ± 1.8 %mc when the uncertainty is given at the 95 % confidence level

($k = 2$). This can be considered a very good result, since the moisture range for five different forest-based biomaterials varied from 14.3 to 68.6 %. However, one should keep in mind here that calibration was carried out by the reference LoD method and the microwave instrument was tuned according to the oven-drying measurement results. Also, perfect calibration samples taken from the same truck loads or piles as the measurement samples were used. On average, the standard deviation of each biomaterial in the three moisture ranges was 0.7 %mc for the microwave measurement instrument and 0.4 %mc for the oven-drying method. Thus, the variation of the measurement results for parallel samples of a single material is smaller to that of the LoD method.

To demonstrate the significance of the good representativeness of the actual measurement samples provided by the calibration samples, the microwave instrument was also used with a calibration carried out using calibration samples collected one to three weeks earlier than the measurement samples from another biomaterial load supplied from a different location. In addition to that, the driest calibration samples had moisture contents slightly below 15 % and the suppliers of two biomaterials (milled peat and chipped small sized trees) changed between the collection of calibration and measurement samples. Despite the supplier change, the peat samples and chipped small-sized tree samples seemed visually similar to the calibration samples. However, such imperfect collection of calibration samples may be possible at power plants. The difference in the moisture measurement results between the oven-drying method and the microwave instrument was in this case 0.1 ± 5.2 %mc on average when the incomplete microwave instrument calibration was applied. This result indicates that without optimal representativeness of the calibration samples, the variation in the moisture readings may be large, while the mean value may stay close to the correct value.

The repeatability tests for the microwave instrument showed that the standard deviation for the measurement repeated five times on single samples taken from all six sample materials at three moisture levels was 0.3 %mc on average. The material-specific and moisture-level-specific repeatability tests are described in Section 7.3. Obviously, the five repeated measurements on the single sample cannot be done for the oven-drying method, because the sample changes (dries) during the first measurement.

7.2. Biomaterial-specific measurement results for the microwave instrument at three moisture levels

This section summarizes the biomaterial-specific and moisture-level-specific microwave moisture measurement results for all the biomaterials.

The left-hand image in Figure 7 shows the performance of the microwave measurement instrument for sawdust samples. Sawdust is typically quite a homogenous material, but surprisingly the largest dispersion of the microwave and LoD differences among the six chosen biomaterials was found for sawdust samples: the mean difference in moisture content between the oven-drying method and the microwave instrument was -0.1 ± 2.7 %mc for sawdust when a 95 % confidence level was applied ($k = 2$). When considering not only different biomaterials but also moisture levels, the difference and standard deviation were largest for dry sawdust samples among all materials and all moisture levels, being -1.5 ± 2.6 %mc, and thus the microwave instrument underestimated the moisture content of dry samples, as can be seen in the left-hand image in Figure 7.

The measurement results for chipped bark waste and chipped pruning residues are quite similar, although the materials are quite different among forest-based biomasses. The results are presented in the middle and right-hand images in Figure 7. The differences between the microwave moisture

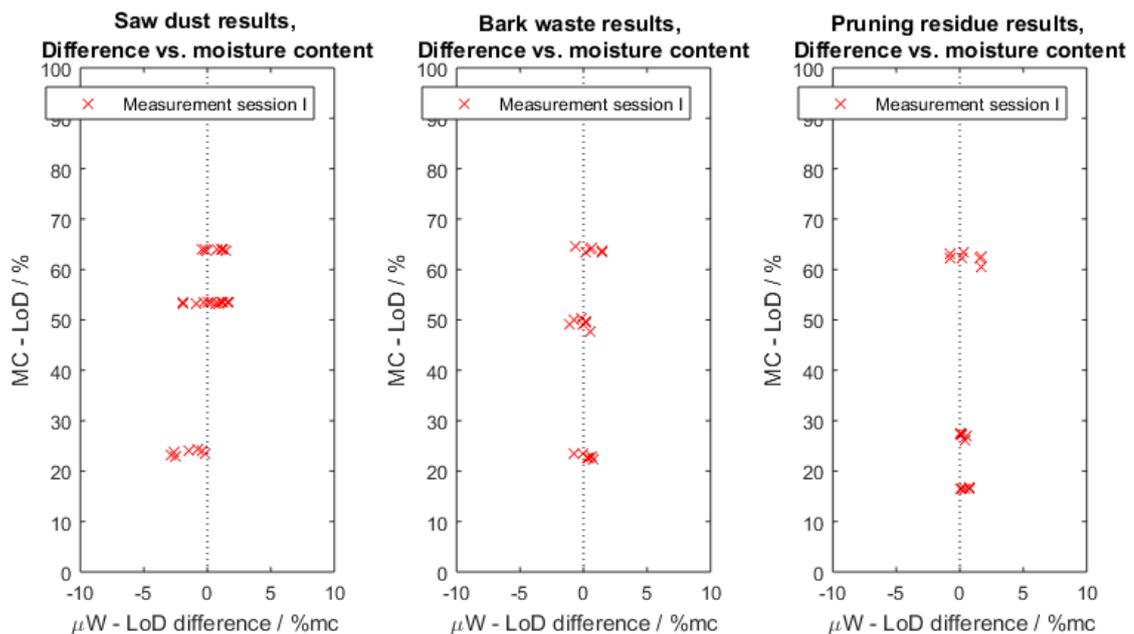


Figure 7. The differences of all measurement pairs between the microwave measurement and the reference LoD method are presented for sawdust (left-hand image), bark waste (middle image), and pruning residue (right-hand image). The x-axis represents the microwave – LoD difference in moisture content and the y-axis represents the moisture content of the sample according to the reference measurement. If the circle or cross falls on the dashed line, no deviation between the microwave and reference instrument measurements exists. The corresponding images of remaining biomaterials are presented in Figure 8.

measurements and LoD measurements are quite small in terms of the mean value and standard deviation, being $0.2 \pm 1.4\%$ mc for bark waste samples and $0.4 \pm 1.4\%$ mc for pruning residue samples, when a 95 % confidence level was applied ($k = 2$).

For chipped small-sized trees, the measurement results are presented in the left-hand image of Figure 8. The difference between the microwave and LoD moisture measurement methods was 0.3 ± 1.9 %mc with a 95 % confidence level. The standard deviation was the second poorest after pure sawdust samples, but it can be recalled from Section 2 that chipped small-sized tree material was mixed with sawdust and this may have affected the results.

The best measurement results for the microwave instrument were achieved with chipped and crushed stump samples. This can also be seen in the middle image of Figure 8, in which all the crosses indicating a measurement difference between the microwave instrument and the LoD method are located close to the dashed zero line. The difference between the microwave instrument measurements and the reference LoD method was -0.2 ± 1.1 %mc when a 95 % confidence level was applied ($k = 2$), so due to the small variation, that mild underestimation can be easily observed in the middle image of Figure 8.

The last tested biomaterial was milled peat and these samples were measured only with the microwave instrument and the reference LoD method, but not with the NMR instrument. The difference between the moisture measurement results achieved with the microwave instrument and the reference LoD method was 0.1 ± 1.5 with a 95 % confidence level. As seen in the right-hand image of Figure 8, the variation of the measurement results for milled peat is quite high for wet samples, but the measurement results for dried peat samples and the peat samples having the moisture content present at delivery are very close to the zero line and thus match the reference LoD method results well.

7.3. Repeatability measurements of the microwave-based moisture measurement instrument

Table 2 summarizes the material-specific and moisture-level-

Table 2. The repeatability test results of the microwave measurement instrument for the six solid biomaterials at three moisture levels. The repeatability test results of the measurement session in autumn 2014 are presented.

Moisture class	Autumn 2014		Autumn 2014	
	Moisture on avg. /%	Std /% mc	Moisture on avg. /%	Std /% mc
	Sawdust		Small-sized tree	
- dried	23.4	0.1	16.5	0.9
- normal	55.3	0.1	53.6	0.7
- moistened	66.4	0.2	68.7	0.5
	Bark waste		Crushed stump	
- dried	23.7	0.2	15.6	0.1
- normal	52.2	0.1	36.2	0.1
- moistened	69.4	0.1	54.8	0.1
	Pruning residues		Milled peat	
- dried	17.5	0.1	25.3	1.1
- normal	32.8	0.1	50.5	0.1
- moistened	71.2	0.1	61.3	0.5

specific repeatability tests of the microwave instrument for single samples. One sample of each sample class was chosen, the microwave moisture measurement was repeated five times on the sample, and the average moisture and standard deviation of the five measurements were calculated. Table 2 shows that the standard deviation of the repeated measurement results varied between 0.1 and 1.1 %mc, depending on the biomaterial. The average of all standard deviations of single samples for different materials and moisture levels was 0.3 %. The highest standard deviation values were achieved with chipped small-sized tree samples and milled peat samples. In Section 7.1, we showed that the standard deviation with parallel samples (not exactly the same) was 0.7 %mc on average; thus roughly more than half of the variation of the microwave instrument results can be explained by the variation of the biomaterial and the rest can be explained by the instrument properties. The repeatability test values from Table 2 are plotted on a graph (see Figure 9,

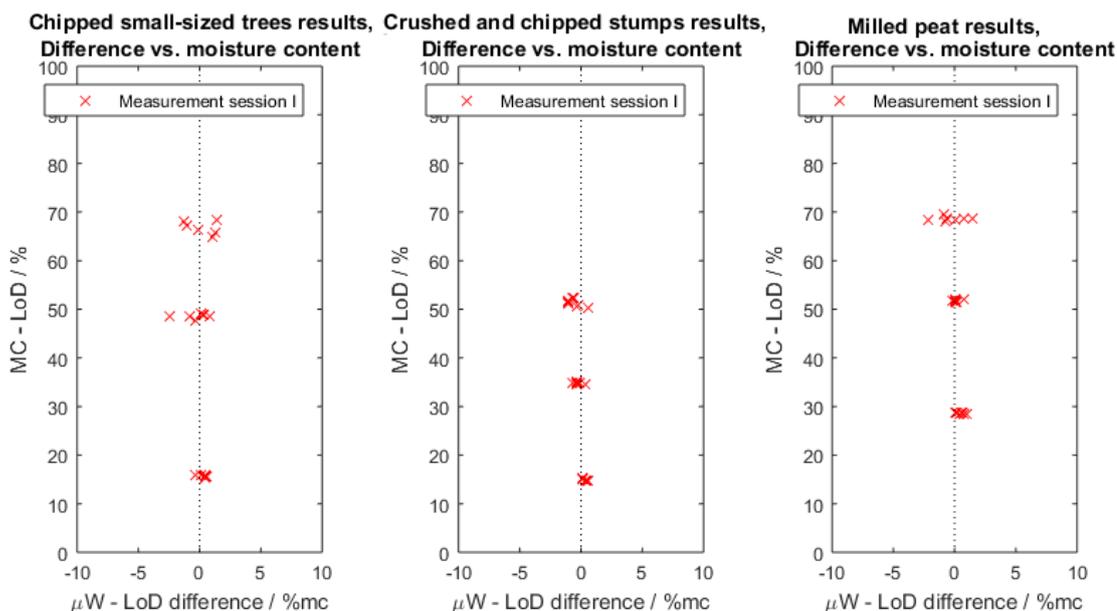


Figure 8. The differences of all measurement pairs between the microwave measurement and the reference LoD method are presented for chipped small-sized trees (left-hand image), crushed and chipped stumps (middle image), and milled peat (right-hand image). The x-axis represents the microwave – LoD difference in moisture content and the y-axis represents the moisture content of the sample according to the reference measurement. If the circle or cross falls on the dashed line, no deviation between the microwave and reference instrument measurements exists.

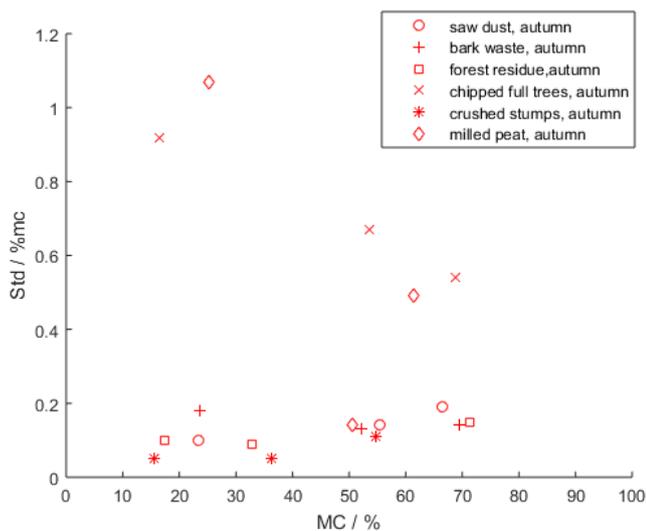


Figure 9: The graph on the right presents the values from the table on the left graphically, and the clear dependence between repeatability and moisture content cannot be observed.

right). A clear dependence between the standard deviation of consecutive measurements of a single sample and the moisture content of the sample cannot be addressed as was done with the NMR instrument, but a dependence between the material type and standard deviation seems to be evident.

8. DISCUSSION

When the two measurement instruments – the Senfit BMA Desktop microwave instrument and the Valmet MR Moisture NMR instrument – were compared, the Senfit BMA achieved slightly better results in terms of both precision and accuracy (0.0 ± 1.8 vs. 1.0 ± 3.8 %mc for all samples on average). However, the good measurement results for the microwave instrument are highly dependent on the calibration procedure. In particular, similarity between the calibration material and the sample material is important for the microwave instrument: if a deviation occurs between the calibration and sample material properties, the measurement accuracy worsens rapidly and the uncertainty may easily be tripled.

The calibration process of the NMR instrument is much more rapid and simpler than for the microwave instrument. The NMR instrument is calibrated only with an empty and water-filled sample container, and the same calibration works for all sample materials. Thus, another moisture measurement method (e.g. the LoD) is not needed for the calibration. The NMR instrument calibration takes about five minutes daily. In comparison, the microwave instrument must be calibrated against another moisture measurement method (typically LoD) separately for each biomaterial and each biomaterial supplier and the calibration process takes at least one day. An appropriate calibration line must always be loaded from memory when the biomaterial type or supplier changes. Thus another (most commonly the LoD) moisture measurement method cannot be neglected with the Senfit BMA Desktop, because it is still needed for the calibration of the microwave instrument. The precision of the microwave instrument is always quite good due to the fact that calibration is carried out according to the reference LoD method measurements. Thus the microwave instrument is tuned towards the LoD reference measurement results during the calibration process.

The measurement range of the NMR instrument is larger than that with the microwave instrument (0–90 vs. 0–70 %). Both the instruments have their own restrictions: the samples for the microwave instrument should be milled to meet the particle size requirements, similarly to the LoD method. The maximum particle size for the NMR instrument samples is not restricted. The samples for the NMR instrument must not include ferromagnetic components and this may occur with, for example, some peat samples. Also, the NMR instrument sample must include at least 20 g of water for a reliable measurement result. This typically corresponds to a moisture content of 5–10 % depending of the mass of the 0.8 L biomaterial sample.

The moisture measurement results showed that the total variation between the moisture content values of parallel samples was slightly larger for the NMR instrument than for the microwave instrument (the standard deviation was on average 0.9 %mc vs. 0.7%mc). Additionally, the repeatability tests of the very same sample showed that on average the uncertainty caused by the instrument was also slightly larger for the NMR instrument than for the microwave instrument (the standard deviation was on average 0.5 %mc vs. 0.3 %mc). The uncertainty caused by the NMR instrument increases when the samples are drier, but this does not occur with the microwave instrument measurements. When observing the average standard deviation of the measurements of parallel samples and average standard deviation of the repeated measurements of the very same sample, as given above, it can be noticed that 0.4 %mc of the total variation explains the variation caused by material properties for both instruments. This was also the average standard deviation of moisture content measurement of parallel samples measured with the LoD method. Based on this result, it can be concluded that the total variation of the LoD method is almost fully explained by material variation. Thus, we obtained the result that the variation caused by the LoD method itself is close to zero, although we cannot carry out the repeatability test for the LoD method by drying the same sample several times.

The variation of the properties between different biomaterials affected the performance of both moisture measurement instruments and the LoD method as well, which was expected.

There is no given measurement accuracy interval for the standardized LoD method in [3], due to the high variability of the properties of different solid biofuels. However, for the solid biofuels having a particle size smaller than 1 mm, a deviation smaller than 0.2 % is accepted for parallel samples [17]. The particle size of all solid biofuels in this research excluding milled peat was larger than 1 mm. The authors held discussions with the key personnel of an enterprise that measures the daily quality of supplied biomasses for a few Finnish power plants with the conventional LoD method. According to them, the standard deviation of 0.5 % for moisture measurement results (i.e. ± 1.0 %-point uncertainty with a 95 % confidence level) is considered as a good result for parallel samples measured with the reference LoD method. They also tested the novel instruments discussed in the article. In industrial use, the commonly accepted deviation between moisture readings of an excellent moisture measurement instrument and the reference LoD method is ± 2.0 %-point with a 95 % confidence level.

The uncertainty interval of the microwave instrument, 0.0 ± 1.8 %, is hardly within the commonly accepted limits for an excellent instrument on average, but the performance varies according to the biomass material: the microwave instrument

satisfies the limits for five materials out of six. The standard deviation of the measurements was outside the range only for sawdust samples. The offset of the microwave instrument measurements was very small due to the fact that this instrument must be calibrated against the reference LoD method.

The uncertainty interval of the NMR instrument, 1.0 ± 3.8 %, was not inside the acceptable uncertainty limits for an excellent instrument on average. Among the five materials researched, the measurement uncertainty of crushed and milled stump material was inside these limits even with the offset. For most materials, the offset was larger in comparison with the microwave instrument. The standard deviation of the measurement results of the NMR instrument was close to the acceptable limits for the three sample materials but not for pruning residue. If the offset is tuned, good enough measurement uncertainty is achieved with the NMR instrument.

9. CONCLUSIONS AND FUTURE WORK

As a conclusion, both the NMR-technology-based Valmet MR and the microwave-technology-based Senfit BMA Desktop moisture measurement instruments might be reasonable alternatives to replace (NMR) or reduce (microwave) the number of the LoD-based measurements, when rapid measurements are needed. However, it seems that neither of them can beat the LoD method in terms of accuracy and precision, but good enough results can be achieved for biomaterial invoicing with the microwave instrument, and the NMR instrument achieves results that are close to the acceptable limits as well.

In future, we will test the performance of our reference LoD method. A few parallel samples have already been measured with an enhanced LoD method [18]. The enhanced oven drying system is equipped with a cold trap to collect water and other VOCs (Volatile organic compounds) from vaporized gases during drying. Tentatively, a small but non-significant effect of VOCs in the moisture measurement results has been observed but this will be reported in more detail in future work.

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