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THE COMPARISON OF A MICROWAVE BASED BIOENERGY MOISTURE MEASUREMENT INSTRUMENT AGAINST THE LOSS-ON-DRYING METHOD

Version 2

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Abstract – This article compares the performance of a microwave based moisture measurement instrument against the conventional moisture measurement reference method Loss-on-Drying (LoD). Six different biomaterials were measured at three moisture content levels with both measurement methods. After instrument calibrations, the difference and variation of the measurement results and the repeatability of the instruments for parallel samples were estimated. Good agreement between the measurement methods was achieved with careful microwave instrument calibration.

Keywords: moisture measurement, bioenergy, microwave, oven drying, LoD

1. INTRODUCTION

The Loss-on-Drying method (LoD) is conventional and still the only standardized method to measure the moisture content of biomaterial load. To determine the moisture content of the load, at least 300 g sample is taken from the biomaterial load of a truck or a 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 standardised LoD method [3] i.e. by weighing the sample and then drying it for several hours in an oven at the temperature of 105 °C ± 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 sample. The LoD procedure is too slow for the efficient process control, but at least in Finland it is still the only moisture measurement method allowed for the basis of payment of the biomaterial delivery lot. In a typical case at a power plant, the moisture content estimate for the biomaterial load is available after two days from delivery.

To overcome the slowness problem of the LoD method, novel rapid moisture measurement methods have challenged the conventional oven drying method. In this research we studied one of these new methods, namely the microwave based moisture measurement solution developed by Senfit Ltd. The microwave instrument must be calibrated using the LoD method, but the measurement takes only a few seconds, whereas the LoD moisture determination takes at least 16 hours. However, new research results are urgently needed for demonstrating the performance of such instruments designed for delivering moisture data to biomaterial invoicing. Only a few publications about commercial biomaterial moisture measurement instruments based on microwave technology could be found during the research [4][5], but they researched the performance of a corresponding microwave instrument with selected biomaterials in restricted moisture range i.e. in the moisture content of delivery.

This research is a part of a European project METefnet [6], which concentrates on the creation of unambiguous SI traceability chain for measurements of moisture in solid materials.

2. BIOMATERIAL SAMPLES AND SAMPLE PREPARATION

The biomaterial samples for the research were collected from the lorries transporting solid biofuel loads into a local power plant. Six different biofuel sections presented in Fig. 1 were chosen:

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

Fig 1. Solid biofuel samples from upper left corner: Saw dust, bark waste, pruning residue, chipped small-sized trees, crushed stumps and milled peat.

The amount of about 50 litres of each selected biofuel section was collected. Each biofuel section was taken from the single location of the same truck load to ensure the similarity of samples for the research. This was opposite to the normal sampling procedure, in which a biofuel sample is collected from several locations from the load to ensure that the sample represents the whole load.

All biofuel sections were measured at three different moisture levels. Thus the 50 L section samples were divided in three parts. The first part was measured in the moisture content of delivery. The second part was spread on a flat pool and dried at room temperature from 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 in the beginning to increase the moisture content of the sample. The amount of the added water was determined and it depended on the moisture content of delivery of each biofuel section. The sample was stored two days in the water tub with a lid. After the two days moistening period the free water was removed from the moisturised biomaterial. Altogether, there were 18 different biofuel sample sets for the research, i.e. six biofuel sections at three moisture content levels.

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

The number of samples was from 5 to 15 pieces for each biomaterial and moisture level depending on the biomaterial, moisture content and calibration samples. Altogether the moisture content of 191 biomaterial samples – 400 g each - were measured in this study using two measurement methods: the microwave based instrument and the LoD reference method.

3. RESEARCH INSTRUMENTS

Four ovens were used in the reference moisture measurements to dry out the biomaterial samples during the measurement session. All the ovens were made by Termaks [7], and they included two pieces of TS4115, and two newer models, TS8056 and TS8136. Single oven can simultaneously dry 6 to 18 biofuel samples of 400 g depending on the oven model and measures of sample containers.

Senfit BMA Desktop [8] was used as the microwave based instrument in this study (See Fig. 2). The moisture content measurement range of the instrument is 0 % - 70 %. The instrument was tuned for a 400 g ± 5 g biomaterial samples placed in a plate-shaped measurement bowl. The bowl is inserted in the measurement chamber behind the lower black lid in Fig 2. The moisture measurement of the sample takes about ten seconds. The particle size of the sample should be no more than 31 mm × 31 mm × 31 mm.

Fig. 2. Senfit BMA Desktop, the microwave moisture measurement instrument on the left and the plate-shaped measurement bowl of the instrument with a crushed and milled stump sample divided in three parts on the right.

4. INSTRUMENT CALIBRATION

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

The careful calibration of the microwave moisture measurement instrument is essential for the reliable measurement results. The calibration of the microwave instrument is made with samples of which moisture content have been determined using the LoD method. Closer the properties of the calibration material are the properties of the sample material, better the measurement results achieved. The calibration of the microwave instrument should be done supplier-specifically for every biomaterial section and prior to the biomaterial moisture measurements. Though the measurement range of the microwave instrument is from 0 % to 70 % moisture content, 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 twice. The first calibration for the

measurement was done for the chosen six biomaterials mentioned in Section 2. According to the instructions of the instrument manufacturer personnel, two samples of each material were dried/moistened to four moisture levels so that there were eight calibration points for each biomaterial sections to create a calibration curve for the microwave instrument. The calibration samples were collected 1 - 3 weeks before the measurement procedure of the research samples. For five materials of the six, the driest calibration samples got slightly below the 15 % moisture content (The final moisture was between 9 % and 12 %) thus the calibration curve was slightly distorted. Additionally, the biomaterial supplier of chipped small sized trees and milled peat changed between the collection of the calibration samples and the measurement session. Probably these decreased the measurement accuracy, in spite of the fact that the measurement samples were visually similar in comparison with the calibration samples.

The second 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 sets and moisture levels after milling (to meet particle size requirements) and mixing the biomaterial. Thus the calibration curve was derived now from six calibration points for each biomaterial (two samples at three moisture levels). After the second calibration, the results were improved, that can be seen later in Tables 1-7 of Section 6.

It should be noticed that only a few moisture levels were used for the microwave instrument calibration: four levels for the first and three levels for the second calibration and two calibration samples for each moisture level with single biomaterial.

5. MOISTURE MEASUREMENTS

Biomaterial samples of ca. 1 kg were stored overnight in plastic bags at room temperature to get constant temperature and moisture content. The measurement procedure of a single sample started by shaking a plastic bag for mixing the sample and the bag was opened then. The plastic bag was emptied on a clean table. A sample of 400 g ± 1 g was weighed and placed in the measurement bowl of the instrument. The bowl was inserted in the microwave moisture measurement instrument. The moisture measurement takes about ten seconds. The instrument measures the sample temperature using IR-sensor and tunes the microwave moisture measurement result based on the temperature of the sample. After the microwave moisture measurement, the biomaterial sample was weighed, placed in a metal container and inserted in the oven for the reference moisture measurement with the LoD method. After each measurement with the microwave instrument, the sample was placed in a drying oven for at least 23-24 hours ensuring at least 16 hours heating at 105 °C to meet the requirements of the standardized oven drying method. Due to the long drying time, several ovens were used to obtain efficient operation. After the drying period, the sample was weighed again and the detected mass loss represents the vaporized moisture from the sample. Finally, the moisture content estimate for the sample was calculated by dividing the mass loss with the initial sample mass.

The repeatability of the microwave instrument was tested by repeating the measurement of a same sample five times. The moisture contents of 18 different biomaterial samples (six biomaterial sections at three moisture levels) of 400 g were measured five times each. The sample was removed and placed back in the measurement chamber of the instrument between separate measurements. At the same time, the sample bowl was turned clockwise roughly 30° so the sample position was always slightly different in the microwave instrument.

6. MEASUREMENT RESULTS

Our results show that good agreement between microwave and LoD measurements were in general obtained when the calibration samples were not below the 15% moisture content and the measurement samples and the calibration samples were from the same supply location. The agreement was less satisfactory when the calibration samples were taken some weeks before the measurement from another truck load. In that case, the biomaterial quality properties derived from growing location, storing conditions etc. may be totally different between the calibration and the measurement samples. This finding is well in agreement with the information provided by the instrument manufacturer.

6.1. Common results of the all samples

As shown in Table 1 the average difference between the moisture results determined with the oven drying method and the microwave instrument is (0.0 ± 1.8) %mc when applied the optimal calibration of the microwave instrument. Here, the uncertainty is given at the 95 % confidence level. This can be considered a very good result, since the moisture range for six biomaterials varied from 14.9% to 68.3%. On average, the standard deviation of each biomaterial in 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 a single material is slightly smaller with the LoD method.

To demonstrate the significance of good representativeness of the calibration samples to the actual measurement samples the microwave instrument was also used with a calibration done with calibration samples collected one to three weeks earlier than the measurement samples from another biomaterial load supplied from different location. In addition to that the driest calibration samples were slightly below 15%. As the lower row of Table 1 shows, the difference of 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 applied the incomplete microwave instrument calibration. This result indicates that without optimal representativity of the calibration samples the variation in the moisture readings may be large, while the mean value may stay close to the correct value. On average, the standard deviation of each biomaterial and moisture range was 0.8 %mc for the microwave measurement instrument and 0.4 %mc for the oven drying method.

Table 1. Standard deviations and the differences between the Loss on Drying method and the microwave moisture measurement for all sample materials. The uncertainties are given at 95 % confidence level (k = 2)

All biomaterial samples	Samples/ pcs	Std of LoD on average /%	Std of μ-wave on average /%	Difference with LoD on average /%
Optimal calibration	127	0.4	0.7	0.0 ± 1.8
Incomplete calibration	163	0.4	0.8	0.1 ± 5.2

The repeatability tests for the microwave instrument showed that the standard deviation for the five times repeating measurement for single samples taken from all the six sample materials at three moisture levels was 0.3 %mc. The material-specific and the moisture level-specific repeatability test are described in Section 6.3.

6.2. Biomaterial-specific measurement results at three moisture levels

This section summarizes the biomaterial-specific and moisture level-specific microwave moisture measurement results for all the biomaterials with both the optimal and the incomplete calibration.

Table 2 shows the performance of the microwave measurement instrument for saw dust samples. Saw dust is typically quite homogenous material, but surprisingly the largest deviation from the LoD in a single reading with the optimal calibration was found with dried saw dust samples: Here, the mean moisture content difference between the oven drying method and microwave instrument was -1.5 %mc. However, still larger deviations were obtained with the incompletely calibrated measurement instrument. Also, the variation of the measurement results for the six chosen biomaterials was largest for saw dust.

Table 2. The microwave measurement results for saw dust and comparison with the reference LoD method.

Saw dust	Samples/ pcs	μ-wave moisture avg /%	Std of LoD and μ-wave /%	Difference of LoD and μ-wave
With optimal μ-wave instrument calibration				
- dried	7	22.2	0.4 / 1.3	-1,5
- normal	13	53.6	0.0 / 1.2	0.3
- moistened	7	64.4	0.1 / 0.7	0.5
With incomplete μ-wave instrument calibration				
- dried	9	21.3	0.5 / 0.8	-2.4
- normal	15	52.2	0.0 / 0.4	-1.1
- moistened	9	62.4	0.2 / 1.1	-1.4

The measurement results for chipped bark waste and chipped pruning residues are presented on Tables 3 and 4, respectively. The differences between the moisture and LoD measurements are quite small in terms of the mean value and standard deviation when optimal calibration was applied.

Table 3. The microwave measurement results for chipped bark waste and comparison with the reference LoD method.

Chipped bark waste	Samples/ pcs	μ-wave moisture avg /%	Std of LoD and μ-wave /%	Difference of LoD and μ-wave
With optimal μ-wave instrument calibration				
- dried	6	23.1	0.4 / 0.2	0.2
- normal	7	49.2	0.8 / 0.7	-0.2
- moistened	6	64.5	0.4 / 0.6	0.6
With incomplete μ-wave instrument calibration				
- dried	8	18.8	0.4 / 0.4	-4.2
- normal	9	50.8	0.8 / 1.1	1.6
- moistened	8	64.3	0.4 / 0.9	0.4

Table 4. The microwave measurement results for chipped pruning residues and comparison with the reference LoD method.

Chipped pruning residues	Samples/ pcs	μ-wave moisture avg /%	Std of LoD and μ-wave /%	Difference of LoD and μ-wave
With optimal μ-wave instrument calibration				
- dried	6	17.0	0.1 / 0.3	0.5
- normal	5	27.3	0.5 / 0.4	0.3
- moistened	7	62.8	0.8 / 0.9	0.6
Incomplete μ-wave instrument calibration				
- dried	8	14.7	0.4 / 0.4	-2.0
- normal	7	30.8	0.5 / 0.7	3.7
- moistened	9	67.9	0.9 / 0.9	5.8

During the collection of chipped small-sized tree material samples we noticed that the chosen biomaterial was not pure, but it was mixed with saw dust and this may affect the results in comparison with pure material. However, the microwave instrument measurement results in Table 5 are very similar with the results of the oven drying method without significant difference, but the variation of the measurement readings is larger, i.e. on the same level with the saw dust readings.

Table 5. The microwave measurement results for chipped small-sized trees comparison with the reference LoD method.

Chipped small-sized trees	Samples/ pcs	μ-wave moisture avg /%	Std of LoD and μ-wave /%	Difference of LoD and μ-wave
With optimal μ-wave instrument calibration				
- dried	7	15.9	0.2 / 0.3	0.2
- normal	7	48.3	0.4 / 1.2	-0.3
- moistened	6	66.9	1.2 / 1.3	0.2
Incomplete μ-wave instrument calibration				
- dried	9	14.4	0.2 / 0.9	-1.4
- normal	9	49.8	0.4 / 1.1	1.2
- moistened	8	65.0	1.1 / 1.3	-1.8

Crushed and chipped stump material contained most soil, and thus weaker measurement results were expected in the results. However, Table 6 shows that the difference with the reference Loss on Drying method was very small and the variation of the measurement was as good as in the reference method.

Table 6. The microwave measurement results for crushed and chipped stumps and comparison with the reference LoD method.

Crushed and chipped stumps	Samples/ pcs	µ-wave moisture avg /%	Std of LoD and µ-wave /%	Difference of LoD and µ-wave
With optimal µ-wave instrument calibration				
- dried	6	15.2	0.2 / 0.1	0.3
- normal	6	34.5	0.2 / 0.3	-0.2
- moistened	8	50.8	0.7 / 0.5	-0.5
Incomplete µ-wave instrument calibration				
- dried	8	12.0	0.2 / 0.3	-2.9
- normal	8	34.9	0.4 / 0.5	0.0
- moistened	10	52.6	0.6 / 0.5	1.4

Table 7 shows the effect of calibration on milled peat samples at three moisture levels. The measurement results of the microwave instrument are rather close to the measurement results of the reference oven drying method throughout the measurement range and the variation is smaller when calibrated with highly representative samples.

The measurement results for milled peat indicate that the variation of the measurement results was slightly larger for the microwave instrument than for the oven drying even with optimal calibration.

Table 7. The microwave measurement results for milled peat and comparison with the reference LoD method.

Milled peat	Samples/ pcs	µ-wave moisture avg /%	Std of LoD and µ-wave /%	Difference of LoD and µ-wave
With optimal µ-wave instrument calibration				
- dried	9	29.1	0.1 / 0.2	0.5
- normal	7	51.9	0.2 / 0.4	0.1
- moistened	7	68.3	0.4 / 1.3	-0.3
Incomplete µ-wave instrument calibration				
- dried	11	31.5	0.1 / 0.4	3.0
- normal	9	53.9	0.2 /0.5	2.3
- moistened	9	67.2	0.4 / 1.6	-1.5

Biomaterials are typically very inhomogeneous and the variation of moisture measurement readings between parallel samples was notable even for saw dust. The standard deviation of moisture readings obtained by the oven drying method varied from 0.0 %mc to the poorest case of 0.9 %mc depending on the sample material and moisture level. The mean of the standard deviations was 0.4 %mc. Correspondingly, the standard deviation of moisture readings obtained by the microwave moisture measurement instrument varied from 0.2 %mc to 1.3 %mc depending on the sample material and the moisture level. The mean of the standard deviations was 0.7 %mc. According to the measurement data, the variation of moisture measurement results is generally larger with higher moisture content for both microwave and the oven drying methods.

6.3. Repeatability measurements of the microwave based moisture measurement instrument

Table 8 summarises the material-specific and the moisture level-specific repeatability tests of the microwave instrument for single samples. It shows that the standard deviation of the measurement results varied from 0.1 %mc to 1.1 %mc, depending on the biomaterial. The average of all standard deviations of different materials and moisture levels was 0.3%, but if milled peat and chipped small-sized tree readings are omitted the average standard deviation would be 0.1%! In Section 6.1 we showed that the standard deviation with parallel samples was 0.7 %mc on average; thus roughly half of the variation of the microwave instrument results can be explained with the variation of the biomaterial and another half with the instrument properties.

Table 8. The repeatability test results of microwave measurement instrument.

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

6.4. Residual moisture in the samples

Typically, an increase of 0.1 %mc in the moisture content was observed when comparing drying times of one day and two days to each other. Milled peat was an exception: for samples dried in room temperature an increase of 0,3 %mc was observed after two days oven drying. An oven drying test of five days was carried out for six peat samples without pre-drying in room temperature. This test showed an increment of 0,4 %mc. However, according to the oven drying standard [3] drying more than 24 hours should be avoided due to increasing error caused by the evaporation of volatile compounds other than water. Supposedly, water is mostly removed from the sample prior many VOCs during drying. Thus the results obtained with 23-24 hours drying were considered as the reference

measurement results for microwave measurement in this research.

7. CONCLUSIONS AND FUTURE WORK

As a summary, the microwave-based moisture measurement instrument Senfit BMA Desktop provided results that were in good agreement of the reference results when the calibration procedure for the instrument was carried out carefully. The difference between the microwave instrument and the LoD reference method was (0.0 ± 1.8) %mc on average and mostly below 1.0 %mc for all the sample materials in different moisture ranges. Our results indicate that if the calibration material differs notably from the measurement samples, the difference – or at least the uncertainty – between the LoD reference method and the microwave instrument increases. It is also important, that the moisture content of the calibration samples is strictly within the measurement range – i.e. between 15% and 70% in this study. The performance of the instruments varies according to biomass type and its moisture content. The variation between parallel biomaterial sample measurement results was quite small in both measurement methods. The standard deviation of the microwave instrument was mostly below 1.0 %mc with optimal calibration. The variation of moisture measurement readings caused by biomaterial inhomogeneity seemed to increase with increasing moisture content of the sample.

As the microwave instrument is currently calibrated with the LoD method, the uncertainty of the LoD method needs to be added to the uncertainty of the microwave instrument readings when using it to determine the moisture content of a biomaterial sample. Thus the microwave moisture measurement instrument can never give more accurate results than the reference method used in the calibration, i.e. the LoD method in this research.

In future further biomaterial samples will be measured with the microwave instrument and the reference oven drying method. The sample properties such as temperature and particle size will be also studied. In parallel with the microwave moisture measurement instrument, a magnetic resonance based moisture measurement instrument has also been tested with comparable biomaterial samples. The research results of the MR-instrument will be published in future.

Additionally we are planning to test the performance of our reference LoD method. Few parallel samples have already been measured with an enhanced LoD method [9]. The enhanced drying oven system is equipped with a cold trap to collect water and other volatile organic compounds (VOCs) from vaporized gases during drying. Small effect of VOCs in moisture measurement results has been observed but this needs to be analysed more in details in the future work.

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