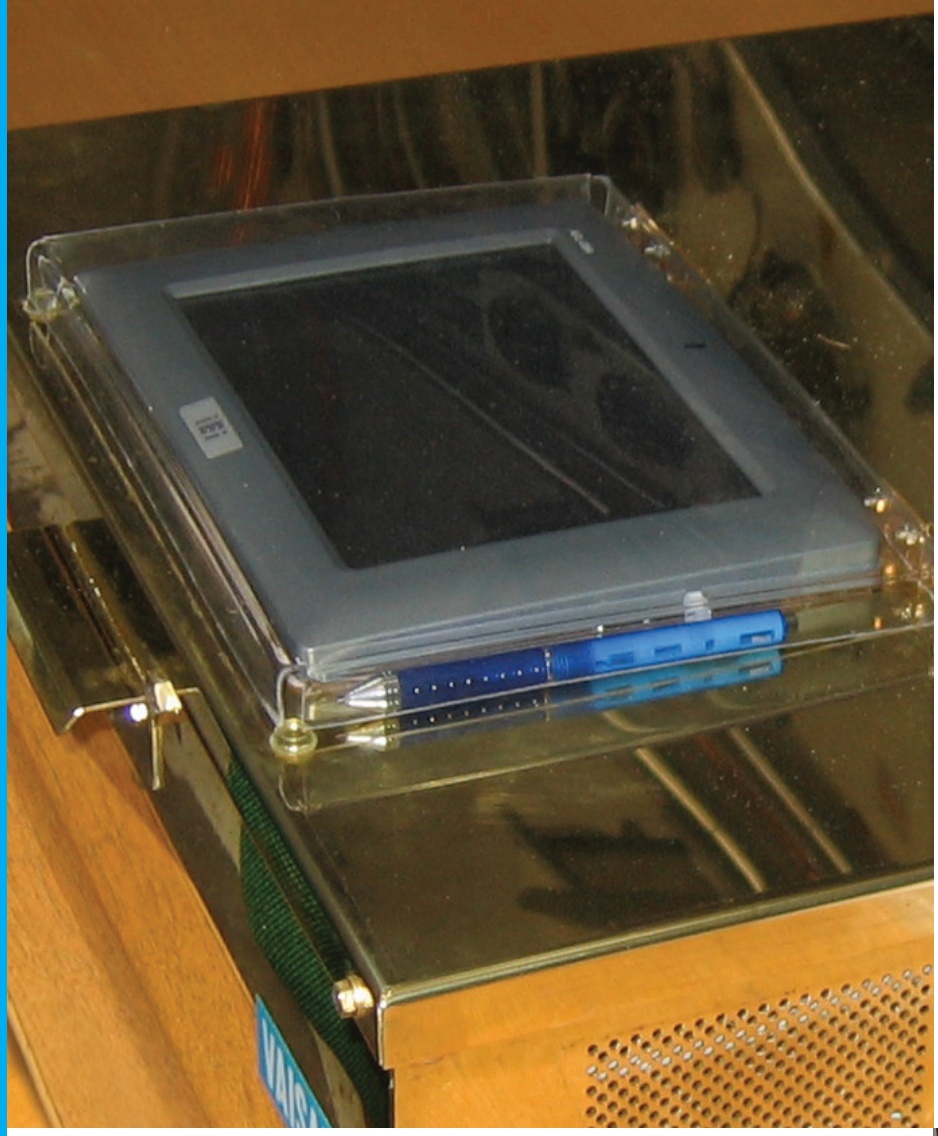

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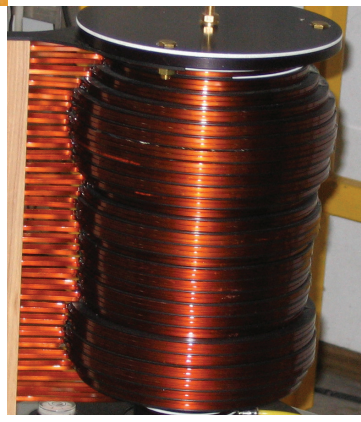
Rapid and accurate biofuel moisture content gauging using magnetic resonance measurement technology



Timo Järvinen

VTT

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Rapid and accurate biofuel moisture content gauging using magnetic resonance measurement technology

Nopea ja tarkka biopolttoaineiden kosteuden määritys käyttäen magneettisen resonanssin mittaukseen perustuvaa laitetta. **Timo Järvinen.** Espoo 2013. VTT Technology 89. 65 p.

Abstract

Biomass is extensively utilised in energy production and as a raw material, such as for the production of liquid biofuels. All those processes will benefit if the moisture content of bio material is known in advance as accurately as possible under transient circumstances. Biofuel trade is increasingly based on the calorific value of fuels. In the first step, this also increases the need for rapid and accurate moisture content determination. During the last few years, large biofuel standardisation has been implemented, emphasising biofuel quality control at all stages of the utilisation chain. In principle, the moisture instrumental measurement can be utilised by many technologies and procedures. Typical techniques are infrared, radiofrequency, micro-wave, radiometric, electrical conductivity, capacitance, and impedance. Nuclear magnetic resonance (MR) and thermal neutron absorption are also applied. The MR measurement principle has been known and utilised already since the early 1950s. It has become the basic instrumental analysis tool in chemistry. It is also well-known as a very accurate method for analysing most compounds, especially substances containing hydrogen. The utilisation of MR metering is expanded extensively to medical diagnostics as a form of magnetic resonance imaging (MRI). Because of the precision of the MR principle, there have for a long time been efforts to apply it in new and different areas, and to make more user-friendly, smaller, and even portable devices. Such a device was designed by Vaisala a few years ago. VTT has utilised Vaisala`s MR prototype for approximately one year for moisture content measurement of different biofuels. The first step in the use of an MR device for moisture determination was the definition of its measurement accuracy compared to the standard method (EN 14774). Those tests proved that the absolute precision seems to be comparable to the standard moisture content measurement method. It was also found out that the MR gauge was the most precise device utilised in the same way, when compared to other alternatives. The gauge was also reliable and easily calibrated. The biggest challenge in using the MR prototype gauge was caused by the volume of sample pots. The average mass of biofuel samples reached about half of the mass presupposed by standard EN 14774 for oven drying. Therefore, at VTT, two separate parallel samples were applied for MR gauging, and the average result was utilised in comparisons and calculations. Already, Vaisala tested the prototype, applying approximately a sample pot twice as big as that used in the prototype, and Metso Automation has recently realised this improvement.

Keywords moisture gauging, MR moisture device, biofuel moisture instrumental measurement

Nopea ja tarkka biopolttoaineiden kosteuden määritys käyttäen magneettisen resonanssin mittaukseen perustuvaa laitetta

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Tiivistelmä

Biomassaa käytetään paljon polttoaineena, ja sen käyttö kasvaa energialähteenä sekä raaka-aineena nestemäisten biopolttoaineiden valmistuksessa. Kaikki nämä prosessit hyötyvät, jos biomassan kosteus tunnetaan etukäteen kaikissa oloissa. Biopolttoaineiden kauppa perustuu yhä enemmän polttoaineen energiasisältöön, joka lisää nopean ja tarkan kosteuspitoisuuden määrityksen tarvetta. Viime vuosina on EU-tasolla toteutettu myös laaja biopolttoaineiden standardointi, joka korostaa laadunhallinnan ja laatutietojen tärkeyttä hankinta- ja toimitusketjussa. Periaatteessa kosteuspitoisuutta voidaan mitata monella instrumentaalimenetelmällä. Tyypillisiä tekniikoita ovat mm. infrapuna (ir, nir), radiotaajuus (rf), mikroaalto, radiometriset, sähkönjohtavuuteen ja kapasitanssiin perustuvat laitteet. Myös magneettista resonanssia (MR) ja termisten neutronien absorptiota on käytetty. MR-menetelmän periaatteet on tunnettu ja menetelmää on käytetty jo 1950-luvulta lähtien. Siitä on tullut paljon käytetty instrumentaalianalyysimenetelmä kemiassa. Se on myös tunnettu tarkkana menetelmänä, jota soveltuu erilaisten yhdisteiden analyysiin ja erityisesti vetyä sisältävien aineiden tutkimiseen. Nykyisin MR-tekniikkaa käytetään spektroskopiassa ja rakenneanalyysissa. MR-teknologian hyödyntäminen on laajentunut lääketieteelliseen diagnostiikkaan magneettikuvauksena (MRI). Jo kauan on pyritty kehittämään pienempiä MR-mittareita. Vaisala Oyj toteutti muutama vuosi sitten tällaisen laitteen. VTT on käyttänyt Vaisala Oyj:n kehittämää MR-prototyyppilaitetta noin vuoden ajan 2011 eri biopolttoaineiden kosteuden mittauksessa. Ensimmäinen vaihe VTT:llä oli määrittää laitteen tarkkuus kosteusmittauksessa verrattuna standardissa (SFS-EN 14774) kuvattuun uunikuivausmenetelmään. Nämä testit osoittivat, että prototyypin tarkkuus oli verrattavissa standardin mukaiseen kosteuspitoisuuden määritykseen. MR-mittaus oli myös tarkempi kuin muut vastaavantyyppiset samalla tavalla käytettävät instrumentaalilaitteet, joita VTT:llä oli ollut käytössä. Prototyyppi oli lisäksi luotettava ja helposti kalibroitavissa. Suurin haaste MR-prototyypin käytössä oli mittausastian koko. Siksi astiaan pystyttiin panemaan tyypillistä biopolttoainetta keskimäärin noin puolet siitä, mitä uunikuivaukseen perustuvassa näytteenottostandardissa (SFS-EN 14774) edellytetään näytemassaksi. Sen vuoksi VTT:n mittauksissa kosteus määritettiin MR-laitteella aina kahdesta samasta näytteestä otetusta osanäytteestä. Laskelmissa ja vertailuissa käytettiin kahden mittauksen keskiarvoa. Jo Vaisalassa prototyypillä testattiin kaksi kertaa suurempaa näyteastiaa, jonka Metso Automaatio on ottanut käyttöön kaupallisissa laitteissa.

Avainsanat moisture gauging, MR moisture device, biofuel moisture instrumental measurement

Preface

Metso Automation gave, at the end of the year 2012, an order to VTT concerning researchers` experience utilising magnet resonance (MR) measurement gauge application in biofuel moisture content determination. The background for the project assignment was that Vaisala had developed a prototype MR device and had given it to VTT to execute a national project dealing with the adaptation of the EN biofuel sampling standard (EN 14778) in Finnish and Nordic ambient conditions. VTT was at the time looking for a rapid instrumental device, enabling fast and accurate moisture content definition, because of a vast number of increment samples presupposed by the CEN adaptation project. In parallel, Vaisala and Metso Automation had a transaction when Metso Automation procured the MR technology developed by Vaisala.

The MR prototype device was used at VTT for about one year in 2011. The biofuels subjected to MR gauging were typical fuels from Finnish forests, such as logging residue chips, but in addition, the moisture content of agro biomass and firewood was determined. The measurements were mostly carried out by VTT´s project group: Timo Järvinen, Antti Heikkinen, and Pilvi Järvinen.

The steering group of the project consists of Metso Automation`s representatives: product manager Lasse Kauppinen, business manager Arvo Rahikkala, director Päivi Tikkakoski, and measurement technology specialist Mikko Haapalainen, and VTT's representatives have been Timo Järvinen, Antti Heikkinen, and Kari Hillebrand.

Timo Järvinen prepared this publication at the beginning of 2013.

On behalf of VTT`s project group, I want to thank Metso Automation for the order of this project, and also Vaisala for allowing us to utilise the new unique instrumental method for biofuel moisture content measurement. At Vaisala, I would like to thank especially Veli-Pekka Viitanen for help and advice.

Jyväskylä 10.4.2013

Authors

Contents

1. Introduction

Biomass, such as biomass from forests and fields, is extensively utilised in energy production and as a raw material, for example in the production of liquid biofuels. All those processes will benefit if the moisture content of the bio material is known in advance as accurately as possible under transient circumstances. This would improve efficiency and usability. On the other hand, it is important to determine the raw material moisture for the control of most processes in the wood industry, such as chemical and mechanical pulping. In chemical pulping, the dosing of digestion chemicals can be more exactly defined if the dry substance content of wood chips is known. In mechanical pulping, the wood chip moisture content gives an idea about raw material processing properties from the fibre quality point of view. The basic question in all cases is similar: the rapid acquisition of precise moisture content data in advance for the process control.

In the energy field, biofuel trade is generally based on the energy content of a fuel in all the Nordic countries. The procedure is also becoming common in other regions of Europe. It presupposes the determination of fuel heat value in fuel receiving. The most important factor influencing the heat value is fuel moisture content. The moisture content should be measured as accurately and rapidly as possible.

Biomass moisture content measurement has been challenging when utilising instrumental methods compared to the other materials, because the moisture (water molecules) is mostly in porous media, such as inside fibres, cell structures, and cells. In addition, moisture is unevenly distributed between the surface and the inside of the material. Therefore, the most utilised method has been, in many countries and in the EU, a standardised oven-drying method (ref. EN 14774). In the standard, the sample of biofuel is dried at a temperature of (105 ± 2)°C in an air atmosphere until a constant mass is achieved, and the percentage of moisture is calculated from the loss in mass of the sample. Depending of the sample quality, drying takes 16 to 24 hours.

Because of the inhomogeneity of biomass, and especially of biomass fuels, sampling is crucial for achieving the right moisture content figure, describing the average moisture of a certain fuel portion. This means that great emphasis has to be put on representative sampling procedures. It has been evaluated that 80% or more in accuracy is due to the sampling, and the rest depends on sample prepara-

tion and moisture content gauging. Therefore, also at EU level, two standards concerning this matter have been implemented during the last few years (2011), among other vast biofuel standardisation processes: EN 14778 Solid biofuels sampling, and EN 14780 Solid biofuels sample preparation. VTT has also very strongly participated in this international standardisation process in the EU, as well as at ISO level.

In principle, moisture instrumental measurement can utilise many technologies and procedures. Typical techniques are infrared (ir, nir), radiofrequency (rf), microwave, radiometric, electrical conductivity, capacitance, and impedance. Nuclear magnetic resonance (nmr) and thermal neutron absorption are also applied. All those methods have been tested and researched at Technical Research Centre of Finland (VTT) during the last 10 to 15 years. As a result, there have been quite many methods that give relatively accurate results for even biomass material like pulp chips or corn, but measuring the moisture content of forest residue chips, for instance, is challenging. Of course, there are accurate methods like nuclear magnetic resonance (NMR) in principle, but the question has been how to make the equipment lighter and more usable in practical work in power plants and mills, and even in fields.

In this context, the nuclear magnetic resonance principle has been used, with the abbreviation MR. This means magnetic resonance measurement analogous to the magnetic resonance imaging (MRI) used in medicine, to avoid confusion with measurements related to radioactive nuclear reactions.

2. Importance and future of instrumental technologies in biofuel moisture content determination

The necessity to develop instrumental moisture measurement is obvious. The need for accurate and rapid gauging of biofuel moisture in power plants and mills increases because the utilisation of biofuels has grown, and increases extensively due to the fact that the EU should produce 20% of its energy from renewable sources, including bioenergy, by 2020. Each member state has its own target; for example, Finland should produce 38% and Sweden 49% of their primary energy consumption from renewable sources by 2020. At the moment, the by-products from forest industries (e.g., sawdust, black liquor) have a high degree of utilisation in both countries. Additional raw materials for energy production include logging residues, stump and root wood, small diameter wood, and other wood not in demand by the traditional forest industries. The target for forest chips in Finland is 25 TWh (13.5 Mm^3) by 2020. The rising quantities and also the types of solid biofuel mean more acquisition and a larger number of contractors delivering biomass, presupposing more extensive biofuel quality control in receiving and processing. At all levels, this emphasises the utilising of automatic and mechanical sampling, and accurate and rapid quantification of biofuel characteristics, including moisture content. In addition, biofuel trade will be based in smaller plants on energy, which means the need to determine at least the fuel moisture content of each load or sub-lot.

2.1 Sampling and sample preparation standards (EN 14778 and 14780)

The Committee for European Standardization mandated by the European Commission has established standards for solid biofuels. Standards for solid biofuels are seen as developing the fuel markets as well as the trans-European fuel trade. The development of standards for sampling and testing of solid biofuels, as well as for fuel quality assurance, assists in the development of the markets for solid biofuels. This helps in reaching the environmental and climatic goals, as well as the

social goals, of the European Commission. Furthermore, competition due to the increasing trade supports keeping the prices of solid biofuels at a reasonable level. Finally, the development of an overall quality assurance system is seen as a key element, because guaranteeing a certain fuel quality is becoming more and more important against the background of increasing regulation of air quality and the goal of using solid biofuels in an environmentally sound way. A solid fuel sampling and sample preparation standard has been realised by work group CEN/TC 335 /WG3.

2.1.1 Solid biofuels sampling EN 14778

The solid biofuels sampling standard describes methods for preparing sampling plans (Fig. 1) and certificates and taking samples of solid biofuels, for example, from the place where the raw materials grows, from the production plant, from deliveries such as lorry loads, or from stock. It includes both manual and mechanical methods. The methods described in this standard may be used, for example, when the samples are to be tested for moisture content, ash content, calorific value, bulk density, durability, particle size distribution, ash melting behaviour, and chemical composition. The main principle of correct sampling is to obtain a representative sample (or samples) from the whole lot concerned. Every particle in the lot or sub-lot to be represented by the sample should have an equal probability of being included in the sample. In order to do this, a sampling plan is needed. The standard also gives instructions on sampling equipment and how to calculate the number of samples needed.

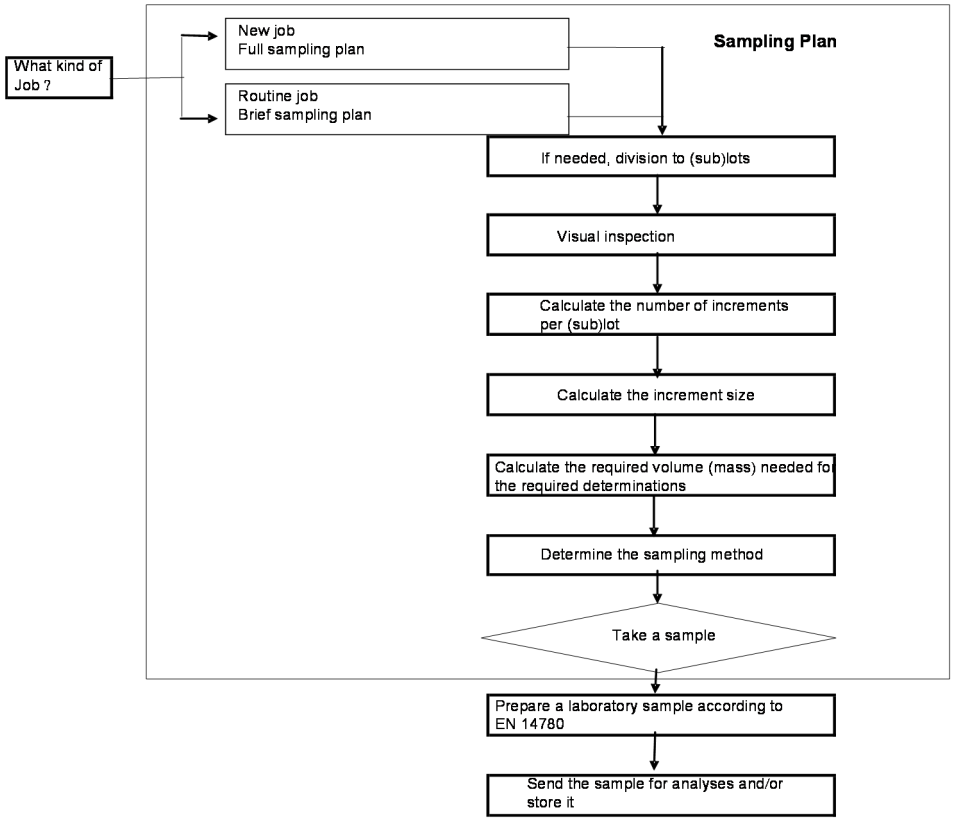

Figure 1. Procedure for sampling (EN 14778).

A lot of attention in the sampling standard has been given to the estimation of the increment numbers based on the sampling precision (P_L). An increment is a portion of fuel extracted in a single operation of the sampling device. P_L is the desired overall precision for the sampling, including sampling, sample preparation, and testing for a whole biofuel lot at 95% confidence level. For precision, recommendations have been given for different fuel types concerning, for example, moisture content. In particular, the recommended moisture content values for “Finnish forest fuels” has turned out to be too tight and can be replaced in a later standard revision stage by more realistic values.

Figure 2. Sampling of forest residue chips.

According to the sampling standard, the increment number is calculated based on Equation 1. Before the calculation, for practical reasons the number of sub-lots (N_{SL}) from which actual sampling will be directed should be determined. The sub-lot is derived from a lot as follows: the lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots, resulting in a possible sample from each. In the case of manual sampling (Fig. 2), a lot may be sampled as a whole only when it is a maximum of 2 500 tonnes, or as a series of sub-lots each of a maximum of 2 500 tonnes, such as for fuel dispatched or delivered over a period of time, a ship load, a train load, a wagon load, or fuel produced in a certain period. Such division into a number of sub-lots can be necessary to:

a) achieve the required precision
b) maintain the integrity of the sample, for example by avoiding bias that can result from the loss of moisture due to standing, or a change of calorific value caused by biological activity
c) create convenience when sampling lots over a long period, such as on a shift basis
d) keep sample masses manageable, taking into account the maximum lifting capacity
e) distinguish different components in a mixture of fuels, such as different biofuel types within one lot (EN 14778).

In Finland, a sub-lot is normally defined as a truck or a tractor trailer load.

$$n_{\min} = \frac{4V_I}{N_{SL}P_L^{\,2} - 4V_{PT}}$$

Equation 1. Calculation of the number of increments.

n = the (minimum) number of increments

P_L= the desired overall precision for the sampling, including sampling, sample preparation, and testing for whole biofuel lot at 95% confidence level

V_I = the primary increment variance

N_{SL} = the number of sub-lots in the lot; in Finland, normally one truck load, when the lot is not divided N_{SL} = 1

V_{PT} = the preparation and testing variance.

Equation 1 can be figured out graphically to give a more illustrative expression for the reader. Figure 3 represents the situation when more realistic values of variances have been used for logging residue chips in different Finnish seasons.

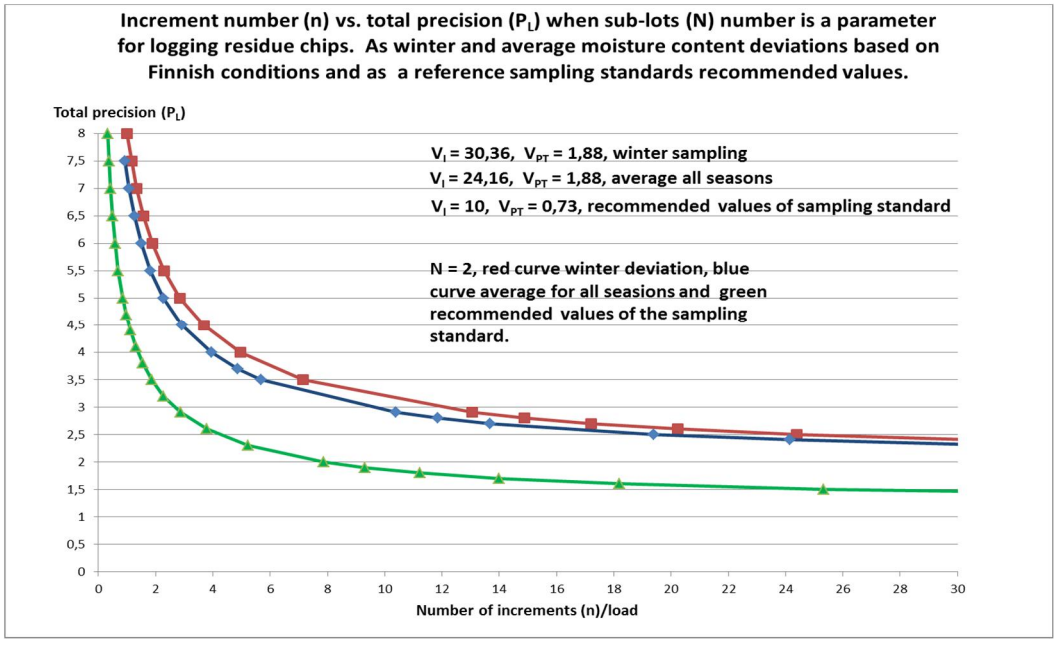

Figure 3. Calculation of increment numbers regarding moisture sampling of logging residues.

Table 1 presents recommended number of increment samples according to the size of forest chip lot based on the more realistic overall precision values in conditions prevailing in Finland.

Table 1. Number of increment samples of forest chips (95 % < 100 mm) based on the sample moisture content at 95 % confidence level when lot size (sub-lot number) grows.

Sub-lot number (load number)	Increment number/load	Increment number/lot	Load size	Over all precision
1 load	10	10	≥ 100 m^3	a. ± 4 %-units
2 loads	5	1	"	n. ± 4 %-units
3 loads	4	12	"	n. ± 3 %-units
4 loads	3	12	"	n. ± 3 %-units
5 loads	3	15	"	n. ± 3 %-units
6 loads	3	18	"	n. ± 2 %-units

Always it has to take at least three increments from a load in spite of a lot would consist more than 6 loads. In the case over all precision increases which is reasonable when considerable fuel deliveries concern.

2.1.2 Solid biofuels sample preparation EN 14780

This standard describes methods for reducing combined samples (or increments) to laboratory samples, and laboratory samples to sub-samples and general analysis samples, and is applicable to solid biofuels. The methods described in this standard may be used for sample preparation, for example, when the samples are to be tested for calorific value, moisture content, ash content, bulk density, durability, particle size distribution, ash melting behaviour, chemical composition, and impurities. The methods are not intended to be applied to the very large samples required for the testing of bridging properties. The main purpose of sample preparation is that a sample is reduced to one or more test portions that are, in general, smaller than the original sample. The main principle for sample reduction is that the composition of the sample as taken on-site shall not be changed during each stage of sample preparation. Each sub-sample shall be representative of the original sample. To reach this goal, every particle in the sample before sample division shall have an equal probability of being included in the sub-sample following sample division. Two basic methods are used during sample preparation. These methods are: sample division and particle size reduction of the sample (Fig. 4). The standard also gives information on suitable apparatus for sample division. A guideline for minimum masses to be retained after each sample division stage, depending on the nominal top size of the material, is given in the standard.

Figure 4. Stump sample particle size reduction and division for moisture content measurement using the MR device.

2.2 Need for rapid and accurate moisture content measurement

Increasing utilisation of biofuels and extending variation of biofuel types mean more samples and, accordingly, more moisture content measurements that are mostly based on the slow and labour-consuming standard method. At the same time, deliveries and contractor numbers are growing rapidly, presupposing delivery control for fuel. In addition, fulfilling the demand for standard sampling and moisture content determination procedures will lead to bigger numbers of samples and determinations. It has been challenging to develop rapid and accurate online measurement systems for biofuel until now, but there are coming solutions based on normal sampling and rapid gauging. The solutions can be called at-line principle working measurements. The rapid measurement makes it possible to increase the number of analysis samples. This means more accuracy, providing the test method is reliable, reproducible, and precise. If these conditions are met, the error in sampling can be reduced, which is the most important factor affecting the representative sampling, sample preparation, and analysis chain when defining the representative average moisture content of an inhomogeneous biofuel lot.

One new alternative for moisture content determination is to utilise the MR measurement method (Fig. 5).

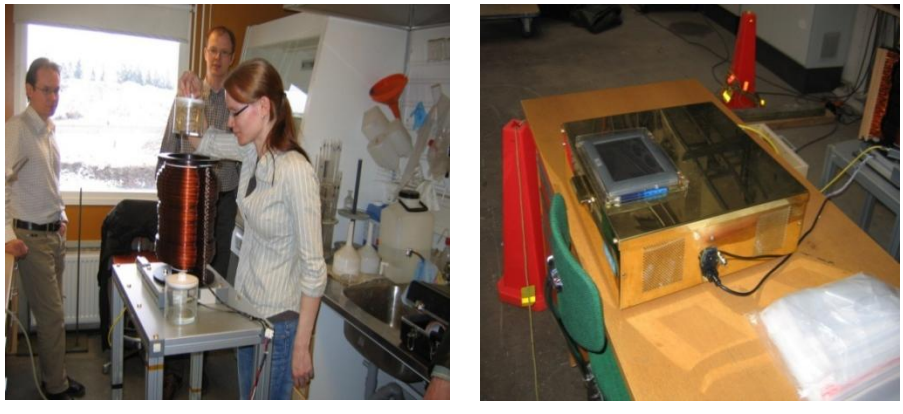

Figure 5. MR moisture gauging device (left magnet, and right measurement and CPU unit).

The moisture result is completed within thirty seconds if the sample is ready in a test pot.

3. Biofuel and biomass moisture content measurements by instrumental methods

Rapid and direct instrumental moisture metering is based on the utilisation of optical methods, like ir/nir absorption measurement, microwave and radiofrequency technology, radiometric methods like utilising x- and gamma radiation and neutron absorption, and even neutron activation analysis (NAA). On big group is to utilise the electrical characteristics of a substance, such as resistance, capacitance, and impedance. During the last few years, nuclear magnetic resonance (NMR) has also been applied to realise simple MR gauges for moisture determination. The basic principle affecting the biomass measurement is whether the sample gauging takes place from the surface or through the material. The surface moisture content dominating methods presuppose quite even moisture content distribution throughout the material. Temperature dependence is critical for nearly all the methods, and gauging sample containing snow and ice rarely succeeds. Another very important factor affecting the usability and precision is how easily the gauge calibration takes place. Mostly, the range of certain similar samples is needed for calibration.

3.1 Optical methods for moisture content measurement

Optical methods are widely utilised for quantitative and qualitative analysis of different materials. Most frequently used is infrared spectroscopy, and the technology depends on the wavelength: nir (near infrared), mir (mid infrared), and fir (far infrared). In (moisture) measurement, common absorption wavelengths are located in the nir range 700–2 500 nm. Figure 6 presents the highest absorption range of liquid water at the nir wavelengths, which are commonly utilised in commercial devices (Järvinen et al. et al. 2007).

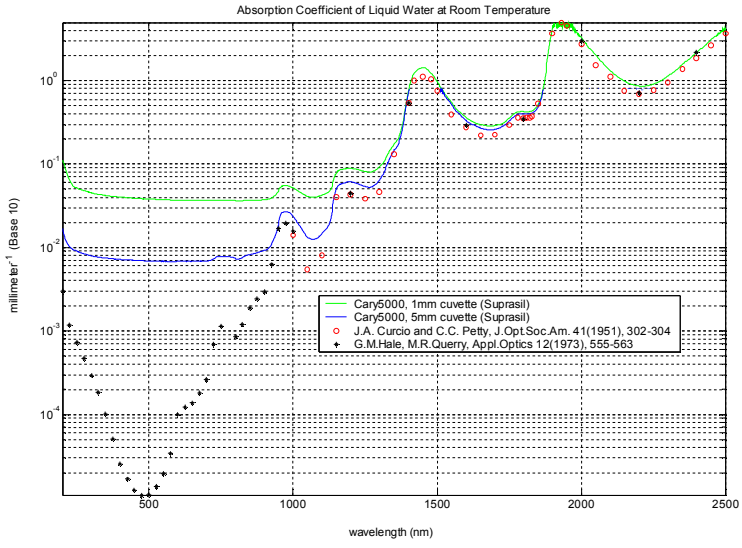

Figure 6. Absorption coefficient of liquid water [mm $^{-1}$] at nir range, based on publications and VTT`s measurement by Cary spectrometer.

Commercial applications typically utilise few wavelengths (2–8), of which one or two actually measure moisture while the rest are for compensation of disturbance. The devices utilise filter discs to separate different wavelengths. Modern tailored moisture content meters are based on array detectors (Fig. 7), allowing the measurement of hundreds of wavelengths at once. The calibration is carried out via complex mathematical modelling (multivariate modelling) (Fig. 8). The target is to make as universal a model as possible helping also to compensate for the surface measurement character of the method. Applying such a model for the prediction of test range moisture gave the results presented in Figure 8. Predicted values follow the moisture change, but there is a systematic error caused by differences in models.

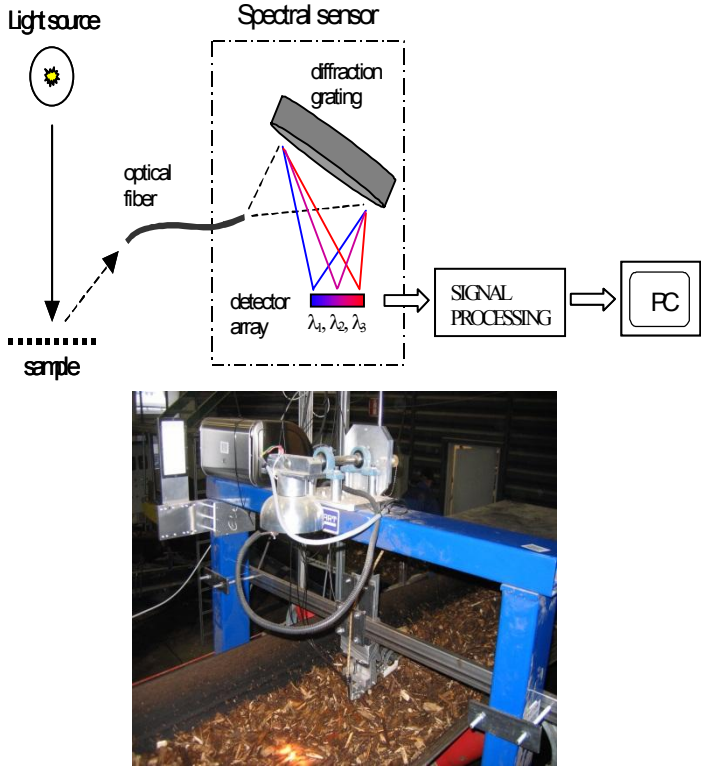

Figure 7. The principle of array detector spectrometer and chip moisture content online-measuring by nir technology.

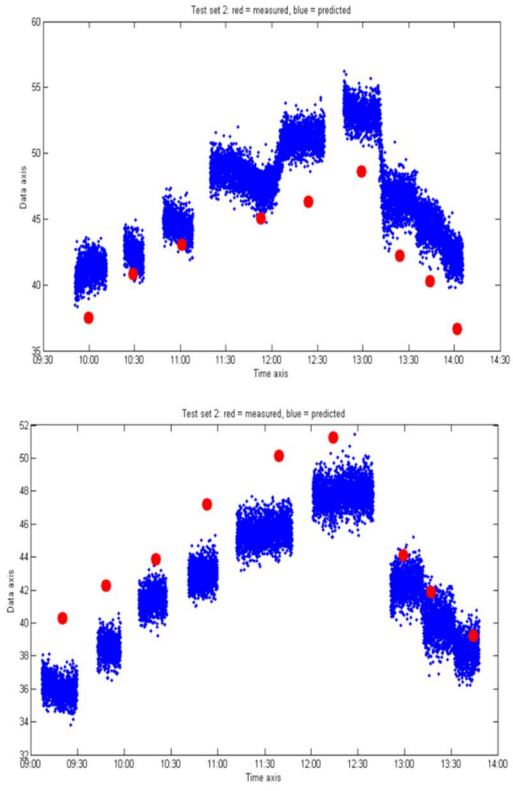

Figure 8. The PLS models testing against a reference range (blue cluster points present predicted moisture and red single points measured reference moisture) (Siikanen 2008).

3.2 Microwave methods in moisture content measurement

The microwave method can utilise the attenuation, phase shift, and resonance sensor for moisture metering. The most common way is to utilise absorption, that is, microwave energy attenuation. Measurement results depend on the sample temperature, which can be easily compensated for. In field gauges, it is also necessary to know the sample density or to measure a certain fixed quantity of a sample. The calibration takes place while measuring the same type reference samples at which the actual gauging will be directed. If the material changes, a new calibration curve will be applied accordingly. Samples containing snow or ice cannot be measured. Microwave gauges are broadly used for biomass (fuels, pulp chip, etc.) (Fig. 9) and, among other things, for coal. The attenuation measurement takes place by the transmission principle, producing the average moisture of the sample layer, whose height or density is known.

Figure 9. Reed canary grass (left) and straw bale (right) moisture content measurement by the microwave principle.

Radio frequency has been studied for moisture measurement of bigger domains. The principle is based on the velocity differences of radio waves, according to the sample (domain) quality. Normally it is calculated as a dielectric constant, for which the dependence of the moisture content is determined. In this way, the calibration curve is established. The measurement takes place after calibration. A transmitter and receiver located in the same box are connected with a domain sample, such as a truck load.

3.3 Radiometric moisture gauging

Radiometric moisture measurement can be based on scattering, absorption, natural activity metering, or excitation. The most common principle is transmission absorption, and excitation in a form of neutron activation analysis (NAA) has also been applied. NAA applications are normally multi-element analysing devices. Dual energy measurement has been applied for coal ash gauging and studied regarding biofuel moisture metering. The dual energy principle can also be applied on a laboratory scale for biomass moisture, but radiation energy levels must be low, leading to thin sample layers.

In principle, radiometric transmission absorption measurements (Fig. 10) describe the material density. The density correlates well with material moisture content if the substance is homogeneous, such as chips for pulping purposes (Fig. 11). If the chip quality changes, the calibration is transformed accordingly. The natural density variation of forest residue chips is, however, so large that a corresponding correlation with reference moisture found in a pulp chip test cannot be observed (Järvinen et al. 2008).

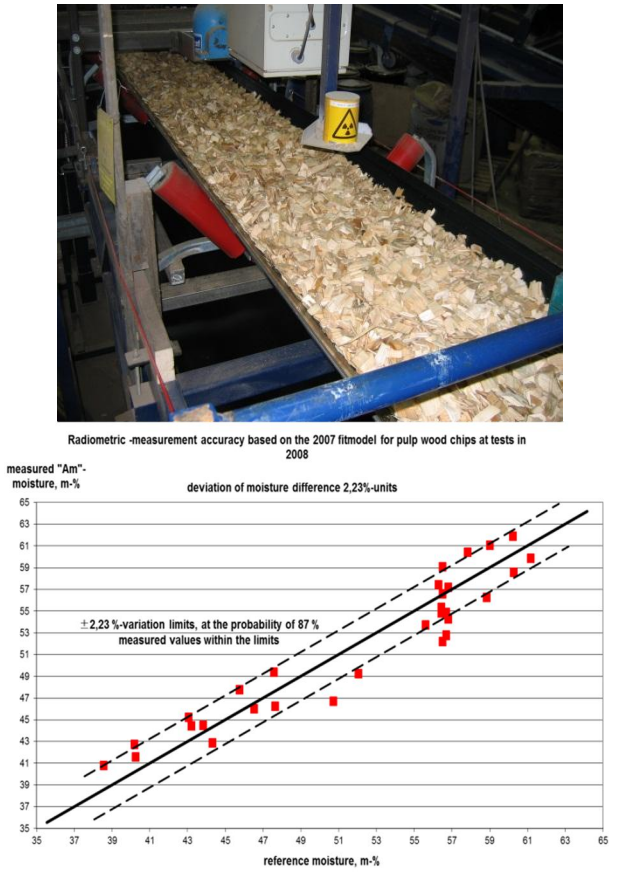

Figure 10. Radiometric density measurement (^{241}Am and ^{127}Cs sources) (left) and moisture metering accuracy when homogenous wood chip is measured (right).

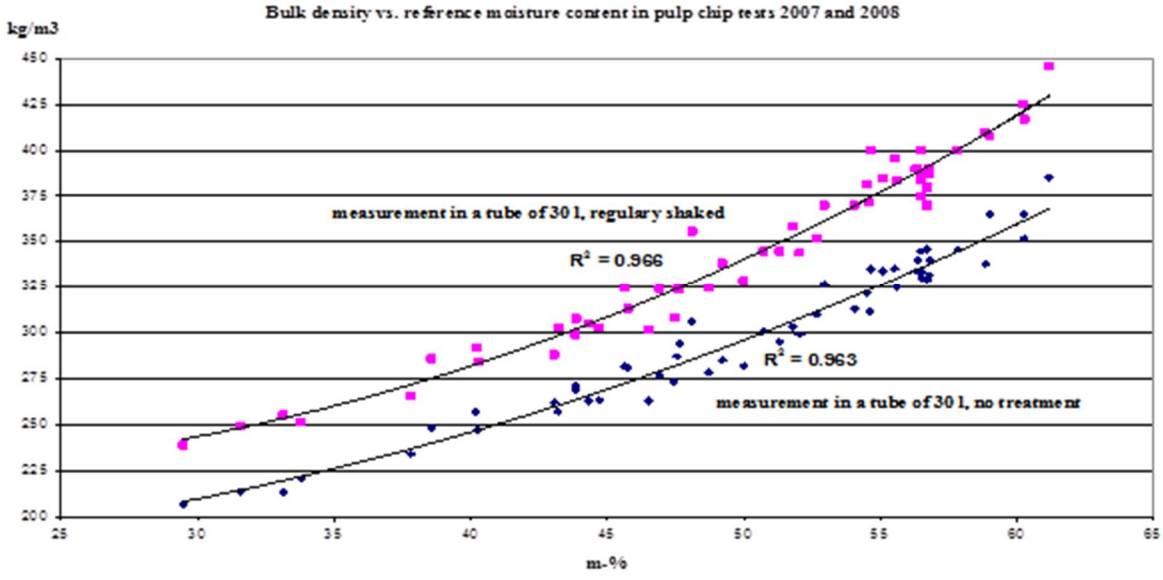

Figure 11. The reference moisture content dependence on the bulk density measurements, based on the sample volume and weight in pulp chip tests of 2007 and 2008.

The radiation absorption measurements, as well as other radiometric measurements, are not very sensitive to temperature change or snow and ice in a sample.

3.4 Electrical methods for biomass moisture measurement

The moisture content of a biomass has an effect on the electrical properties over nearly the whole frequency range. The most common methods of measuring moisture content are based on the resistance or dielectric properties of a sample. There are many kinds of hand-held gauges available for moisture measurement utilising those principles. Instruments that use the resistance measurement principle are suitable for a narrow moisture range, generally below 30 w-%. They are sensitive to temperature change, and density variations must be compensated for.

Under “dielectric properties measurement”, there is a wide range of gauging types. Moisture measurements using dielectric methods are possible in the radio frequency (RF) or microwave range. During calibration, the measured values of attenuation, phase shift, or frequency shift, and so on containing the complex value of permittivity will be assigned to the moisture content determined by the oven dry method. The measured complex value of the relative permittivity will be influenced by the moisture content, but also by density variations, ionic conductivity, and temperature. One general name for the gauges is capacitive meters, which

utilise only a few or one frequency. These gauges also have limitations regarding moisture content distribution in a sample: the surface moisture easily dominates the metering results. To avoid the limitations of material dielectric properties measurement, it has been applied to impedance spectral metering. Impedance spectroscopy has measurement range corresponding to the capacitive method, but it utilises several frequencies instead of one. In this method, subject matter can be described by electrical models, enabling analysis of specific material properties. This technique enables the use of more sophisticated mathematical modelling for the analyses, such as chip moisture content under variable conditions. Model tools are comparable with nir multivariate models. The sensor element must have close contact with the material to be measured. An air space between the sensor and the material surface prevents the measurement. The method is sensitive to temperature change, and samples containing snow or ice cannot be measured. There are some portable devices available on the market, but in general, the method is still under development (Fig. 12).

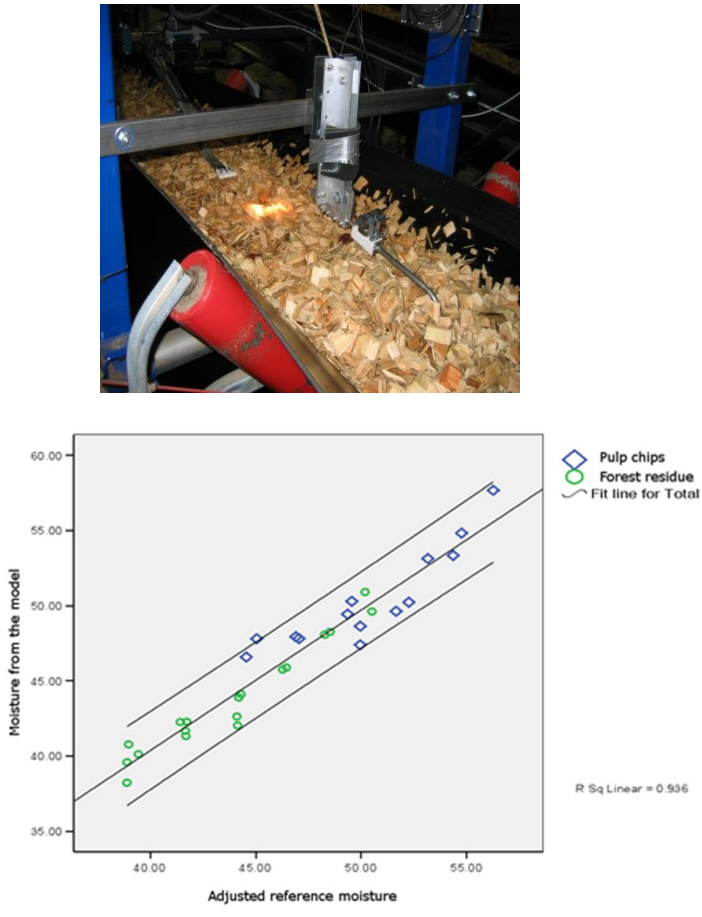

Figure 12. Two impedance sensor heads gauging pulp chip moisture (left) and measurement accuracy compared with reference moisture content (right) (Tiitta 2008).

4. Theoretical background of magnetic resonance measurement (MR)

The first nuclear magnetic resonance experiments were performed as early as 1945 (Bloch and Purcell). The phenomenon was utilised firstly for accurate measurements in laboratories. Much later, it was realised for use in non-destructive testing and measuring. At the moment, nearly all chemical departments in universities all over the world have NMR devices especially for (structural) compound analysis, and numerous hospitals utilise NMR imaging or magnetic resonance imaging (MRI) for medical diagnostics on patients.

The method is based on the phenomenon that an atomic nucleus (spin ≠ 0) has a nuclear magnetic moment. If the nucleus is placed in a magnetic field (B), its angle (ω) or precession movement is in relation with the field and can be expressed according to the Larmor equation (2).

$$\omega_L = \gamma B$$

Equation 2, where γ = magnetogyric ratio.

The most sensitive nucleus regarding the γ coefficient, that is, with a high and strong value, is the hydrogen proton. The hydrogen proton has a γ value of 42.6 MHz/Tesla. If the static field B = 1 Tesla, then ω = 42.6 MHz. If B = 4.7 Tesla, then ω is about 200 MHz (Ruan and Chen 1998).

NMR spectroscopy is based on the interface between the external magnetic field and the nuclear magnetic moment. If the sample is in an external magnetic field and it is exposed at the same time to electromagnetic radiation, it absorbs energy at a certain frequency equalling nuclear precession movement. This phenomenon is called nuclear magnetic resonance (NMR), which can be measured. Well-known important nuclei that can easily be detected, because they have a high γ value, are ^{1}H, ^{19}F, and ^{31}P. In general, NMR can be best applied for the analysis of substances containing lot of hydrogen. If the material conducts electricity well, it is difficult to create a sufficient effective RF field inside the matter, which prevents the measurement. Molecules, electrons, and ambient things also create small local magnetic fields causing (disturbing) resonance signals in the NMR spectrum (Fig. 13).

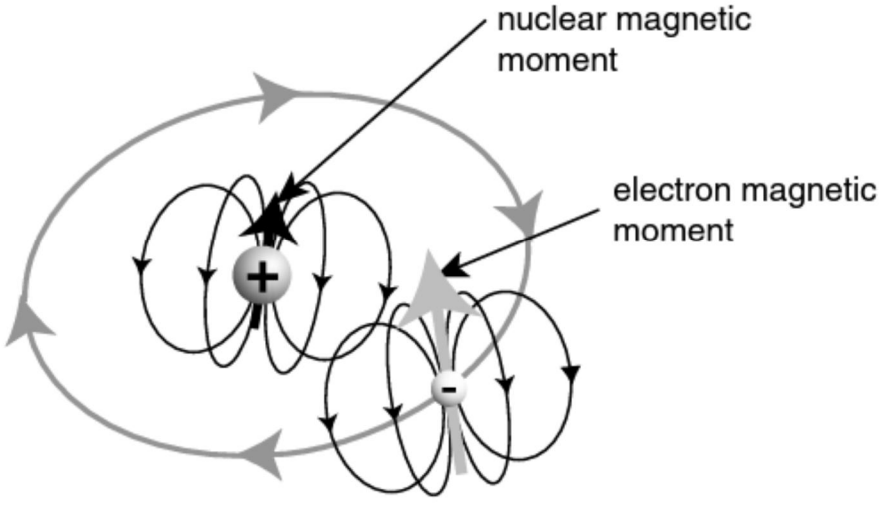

Figure 13. Schematic presentation of a hydrogen proton and electron magnetic moments (Tulkki 2009).

The NMR moisture measurement device consists of a magnet, the magnet coil and enclosure, a pot for carrying the product to be measured, an RF coil that sends and receives the RF pulses for making the NMR measurements, an RF pulse generator, an RF receiver, and a computer that handles the spectral information and analyses the data (Fig. 5). When designing the NMR device, one of the most import factors is how intensive and even the application needs the magnetic field to be. The more effective the magnet is, the more expensive and bigger the actual device will be. For accurate measurement, intensive magnetic fields are utilised, created, for example, by superconductive magnets and high-resolution spectroscopy applications.

5. Applications of MR technology in science and technology

Basic NMR technology is mostly utilised for analysing compounds containing hydrogen and also for studying other NMR-sensitive nuclei containing substances. It is nowadays one of the basic spectroscopic instruments of a chemical research laboratory. There are numerous articles describing NMR utilisation in normal chemical analysis.

It was discovered quite early that an RF signal sent to the sample can be pulsed and the NMR spectrum processed with Fourier transformation. The Larmor frequency is proportional to the field strength and it is a function of position. This makes it possible determine a relationship between the frequency and spatial position of the spins. At present, NMR is utilised especially in spectroscopy, because there is a lot of information in a sample spectrum, which different time-dependent interactions between various nuclei cause.

NMR spectroscopy is a direct tool in chemistry for identifying the structure of both pure compounds and mixtures. It can be said that structure determination for almost all organic and biological molecules begins with NMR spectroscopy. Generally, an ^{1}H NMR experiment reveals in a few minutes whether it is worth continuing or not. If it is possible, there are many different NMR measurement techniques to be selected. NMR is also a versatile spectroscope for the study of the statics and dynamics of condensed matter. In particular, the analysis of chemical shielding and spin-spin couplings is a field of high resolution spectroscopy. Accordingly, there are many types of NMR devices available on the market for laboratories. Actually, already more than ten years ago, nuclear magnetic imaging (MRI) was developed at a commercial product level, due to the development of effective computer-based gradient and image processing technology originating from the field of computer topography (CT) scanning.

MRI is an extension of NMR spectroscopy. MRI provides spatial information about spins. It is a primary instrument in the clinical area of diagnostics of injury or diseases associated with anatomical and pathological changes of organs and tissues (Fig. 14). At present, MRI is also used in biological and food science, providing a new way to get information from bio-based substances (Ruan and Chen 1998).

The ability to flip the magnetisation through the RF pulse allows the manipulation of magnetisation of the nuclei, giving the possibility to characterise the nuclei. For instance, in a mixed phase system, free and bound water and solid can be separated through the relaxation time constants: a very short one for solid, a longer one for bound water, and an extended one for free liquid. The relative magnitudes of the components reflect the quantities of mixed phase constituents. Using the relaxation phenomenon, it is also possible to determine density, image contrasts, and diffusion parameters.

Many industrial research applications are based on relaxation time characterisation, and also NMR spectrum analysis. Several authors have published moisture content profile studies using MRI in fields like wood, food, and the paper industry. However, it is still challenging to achieve online measurement applications in process industry transfer lines, including full-scale belt conveyors. There are already applications for small pipes, but not on a large scale. In MRI technology, full-scale measurement is commonplace in medicine; a patient's whole body will be carried into the magnet drum and, for instance, an accurate image of the patient`s head will be available in a few minutes.

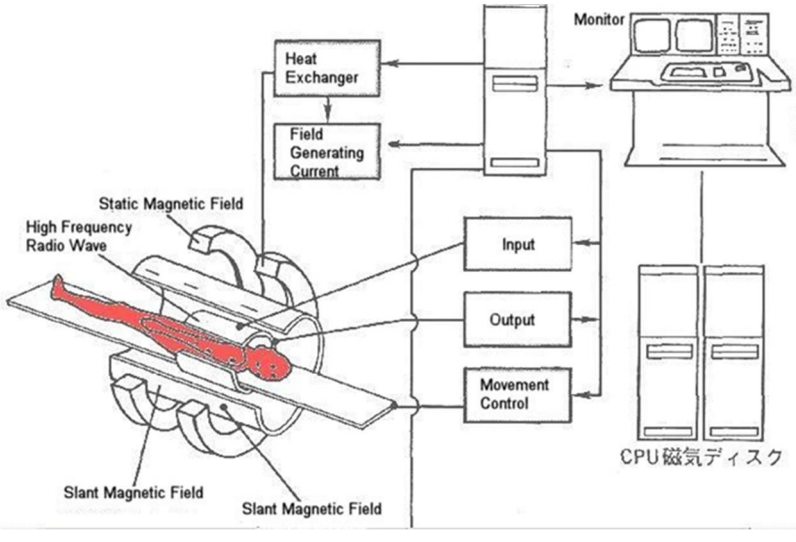

Figure 14. MRI scanner (universe-review.ca/R10-22-tomography.htm).

6. Applying MR technology in moisture content gauging

Basically, MR devices at universities can easily be applied to moisture content gauging of any substances within the RF field that can be created. Actually, a very large range of sample moisture content, and also a tiny one can be defined. This is because of effective magnets leading to intensive and even magnetic fields and a high-resolution spectrum. However, the sample quantities are small, and university equipment is heavy and complex.

Conventional NMR requires a laboratory electro- or superconducting magnet. Spectroscopic information is obtained by using uniform magnetic fields. The technique is inherently invasive because the field uniformity is restricted to small volumes, and materials have to be placed inside the magnet system. Alternatively, inside-out NMR uses open magnet designs for measurements in the field, without sample size restrictions. The trade-off for non-invasive evaluation is reduced sensitivity and lack of resolution due to the non-uniform polarising fields associated with the open magnet configuration. Material assessment is therefore achieved via measurable relaxation times and relative signal amplitude (Prado 2001). This technology is applied to monitor moisture content in wood specimens and to quantify cement hydration, as two potential applications of the hand-held NMR sensor.

The measurement of the moisture content during the drying of wood using the NMR technique has been the subject of several studies since the beginning of the year 2000. Casieri et al. (2004) used a mobile NMR probe as a non-destructive and non-invasive tool for water content analysis on wood samples. They say that the method can be applied to any kind of porous media that has no detectable proton signal from the rigid matrix as, for instance, in building materials. In wood, where a proton signal can be detected from cellulose and others macromolecular components, some considerations and artifices are proposed for eliminating this contribution. The method has enabled the performance of moisture volume fraction analysis on wood samples characterised by different wood species, cutting, and moisture content. According to them, the NMR data on moisture detection as a volume fraction have successfully been compared with those obtained by the gravimetric method.

Anders Rosenkilde and Paul Glover (2002) have applied MRI technique to measurement of the moisture content profile in the surface layer of Scots pine (Pinus sylvestris) sapwood. They used a high-gradient permanent magnet for the

measurement of the moisture profile in the wood during the drying process. The depth resolution reached was better than 20 μm. The paper demonstrates the possibility of measuring the moisture content depth profile in the surface layer from a raw state down to 4% moisture content without removing the wood sample from the apparatus. The main advantage of the applied technique and apparatus was a high resolution over a large field of view. It was possible to measure the moisture content in the wood surface layer during drying up to a depth of 300 μm. A single moisture content profile may be obtained in less than 5 minutes.

Barale et al. (2002) applied the NMR technique (device) to a pulp chip moisture content measurement developed in the Lawrence Berkeley National Laboratory (LBNL). In LBNL, a bench scale sensor was developed that measures the water content of wood chips, brown stock, and black liquor. Their report describes a water content measurement device that uses a permanent magnet. Moisture content measurements were made using ordinary hydrogen NMR at 20 MHz, using a magnetic field strength 0.47 T. According to the results, the moisture content measurements could be made accurately using NMR. It appeared that NMR measurements of moisture content may have been as accurate as measurements made using the TAPPI standard test for moisture content. NMR moisture content measurements can be made rapidly in a few seconds.

7. Portable (mobile) MR gauges for biofuel and biomass moisture content definition

Even though the MR technique has been proved to be an accurate method of measuring the moisture content of biomass, there are only a few attempts to develop mobile versions for MR gauging. There are some tailored gauges described, for example, by Casieri et al. (2004) and Prado (2001) (hand-held sensor head).

The first real step towards a mobile device was taken by Quantum Magnetics, Inc. (QM), a wholly-owned subsidiary of GE Security (Magnuson 2004). The US Department of Energy (DOE) financed QM under the programme Industries of the Future (IOF), a project to investigate roles for low-cost nuclear magnetic resonance (NMR) technology for industrial process and quality control. It was noticed that low magnetic fields, achievable at low cost, lose the ability to obtain spectroscopic information. However, measuring the time constants associated with the NMR signal, called NMR relaxometry, gives indications of chemical and physical states that are of interest to process control and optimisation. Therefore, the goal of this project was to investigate the technical and economic feasibility of using such low-field, low-cost NMR to monitor parameters, enabling greater process efficiencies. To create the magnetic field, a permanent magnet was utilised with an RF coil solution (Quantum Mechanics Inc.), and amplifiers were developed further (VILNAD, i.e. variable-impedance low-noise amplifier design) (Magnuson 2004). The actual device can be seen in Figure 15.

Figure 15. Light MR device (on the table: magnet, RF coil, amplifier; on the floor: RF receiver, PC, and power supply). Cable length between magnet and CPU: 6 m (Magnuson 2004).

Laboratory testing demonstrated that the MR system was capable of accurate (± 0.5%) measurements of the moisture content of wood for moisture ranging from 2% to more than 140% (referenced to the wood's dry weight). Accuracy exceeded that offered by existing instrumentation when the moisture content was in excess of the fibre saturation point (~20%). Accuracy was independent of the wood form: solid wood, wood chips, or sawdust.

The second attempt to develop a mobile MR gauge was made by Vaisala Inc. about five years later than QM. Vaisala´s device utilises electromagnet and lighter RF sending and receiving systems, as well as a data processing arrangement available on the market at the time. Figure 16 presents the gauge.

Figure 16. Vaisala MR moisture gauging device (CPU in front and electromagnet on the left; behind and near the wall are sample pots, which will be put into the magnet for the measurement.).

Vaisala stated the MR gauge accuracy as ± 2 w-% (sample total mass) at a certain range of biomass moisture content. When the sample moisture content is below 10 w-%, the accuracy weakens.

8. Moisture content determination according to the EU standards (EN 14774)

EN 14774 contains three parts, 1–3: Part 1: Total moisture – Reference method; Part 2: Total moisture – Simplified method, which is mostly utilised; and Part 3: Moisture in general analysis sample. Below is described brief description of all three parts (http://www.solidstandards.eu/images/modules/solidstandards_module-general_eng.pdf).

EN 14774-1:2009 Solid biofuels - Methods for the determination of moisture content – Oven dry method – Part 1: Total moisture – Reference method

This EN standard is applicable to all solid biofuels and describes the reference method for determining the total moisture content of a sample by drying in an oven. It should be used when high precision of the determination of moisture content is necessary. A sample with a minimum mass of 300 g is dried at a temperature of 105 ± 2°C and in which the air atmosphere changes between 3 and 5 times per hour, until constant mass is achieved. The moisture percentage is calculated from the loss in the sample mass. The procedure for the correction of buoyancy effects is included in the method. The dried sample has to be weighed while still hot, which gives a buoyancy effect that has to be compensated for when the highest precision is required. The apparatus, sample preparation, procedure, and calculation are described.

EN 14774-2:2009 Solid biofuels – Methods for the determination of moisture content – Oven dry method – Part 2: Total moisture – Simplified method

The principle of this EN standard is similar to EN 14774-1, and it may be used when the highest precision is not needed, such as for routine production control on site, which means for most analysis. The only difference compared to Part 1 is that there is no buoyancy compensation in Part 2. A sample with a minimum mass of 300 g is dried at a temperature of 105 ± 2°C in an air atmosphere until a constant mass is achieved, and the moisture percentage is calculated from the loss in the sample mass. The apparatus, sample preparation, procedure, and calculation are described. Figure 17 presents a typical oven and samples in containers of at least 300 g each.

Figure 17. Typical sample drying oven and sample containers.

EN 14774-3:2009 Solid biofuels – Methods for the determination of moisture content – Oven dry method – Part 3: Moisture in general analysis sample

This EN standard is applicable to all solid biofuels, and it describes the method for determining the moisture in an analysis sample by drying the sample in an oven. It is to be used for general analysis samples described in EN 14780. A general analysis sample is defined as a sub-sample of a laboratory sample, with a nominal top size of 1 mm or less and used for a number of chemical and physical analyses. The analysis sample is dried either in an air atmosphere or in a nitrogen atmosphere at a temperature of 105 ± 2°C, and the moisture percentage is calculated from the loss in the test sample mass. The apparatus, sample preparation, procedure, and calculation are described. A minimum of two determinations shall be carried out on the test sample.

9. Comparison of standard measurement with MR results

VTT has utilised Vaisala`s MR prototype for approximately one year (in 2011) for moisture content measurement of different biofuels. The first step in the use of an MR device for moisture determination was the definition of its measurement accuracy compared to the standard method (EN 14774). Accuracy testing of the MR device was carried out by first measuring the sample moisture content using an MR gauge and immediately after that setting the same sample in an oven according to the standard (EN 14774). The testing method is possible because the sample characteristics, especially concerning moisture content, do not change during MR measurement, which takes a few seconds. The quantity of energy directed at the sample in MR gauging is so minimal that it cannot be observed through normal temperature measurements.

Standard (EN 14774) requires a sample size of at least 300 g for oven drying. The sample pot volume of Vaisala´s MR prototype was 630 ml (Fig. 18). This means that a 30–60 w-% moisture content range can be MR measured for approximately 150–200 g biomass-based fuels. Therefore MR measurement was carried out on two samples (together > 300 g) to achieve a sufficiently large sample for oven drying, as required by the standard. In comparison to the single standard results, a weighted average of two NMR measurement results was used.

Figure 18. Standard sample container (~ 300 g) and two MR sample pots (~ 2 x 150 g).

The determination of MR accuracy was carried out with different types of wood chips (Figs. 19 and 20), fire wood pieces (Fig. 21), and Reed canary grass (Fig. 22).

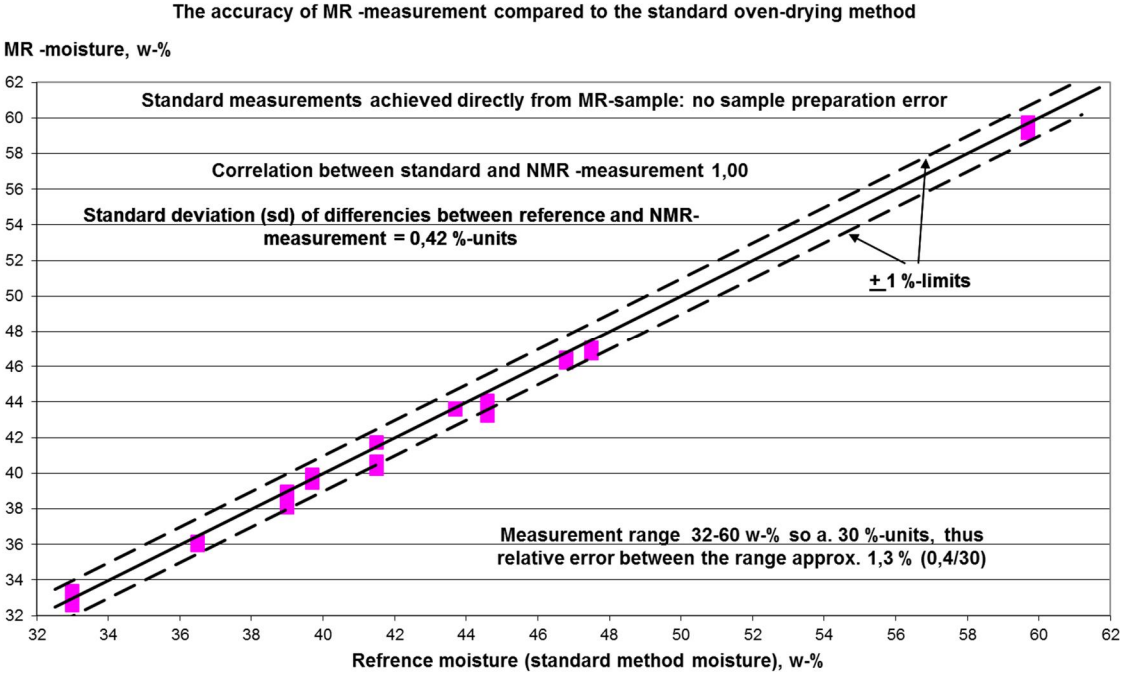

Figure 19. The accuracy of MR measurement compared to the standard oven-drying method with whole tree chips (including stem and branches).

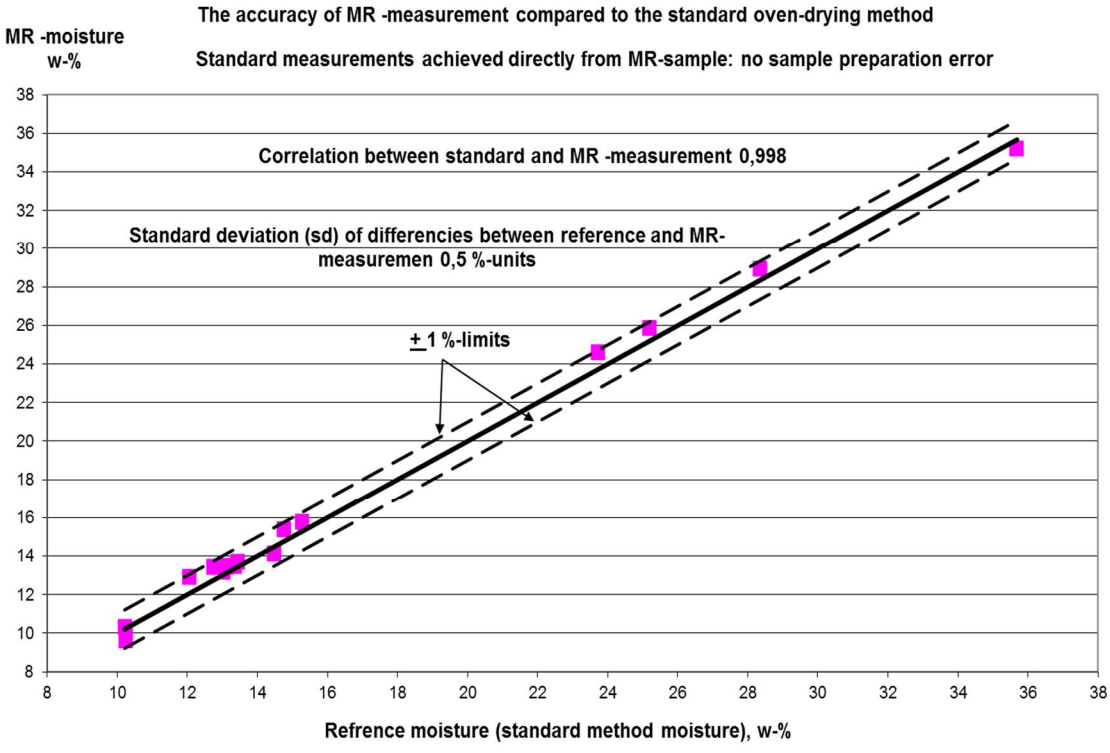

Figure 20. The accuracy of MR measurement compared to the standard oven-drying method with small tree chips (stem wood chips including bark).

As can be seen in Figures 19 and 20, the uniformity of MR measurement with standard moisture content determination is very good on a wide range of sample moisture content when different types of wood chips are measured. The wood material was also measured by MR in the form of one wood piece, filling an MR sample pot. Pieces were cut from chopped firewood with different moisture content (Fig 21).

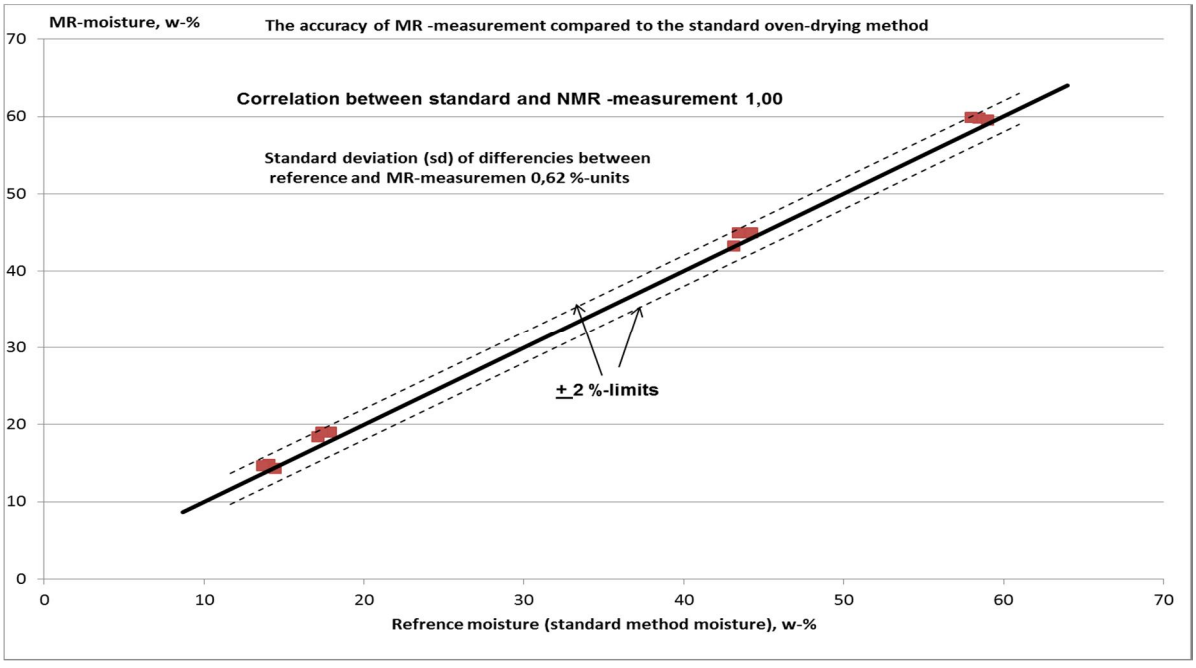

Figure 21. The accuracy of MR measurement compared to the standard oven-drying method with chopped firewood pieces.

In Figure 21, it can be seen that there is slightly lower accuracy compared to the wood chip measurements, but still at a very satisfactory level. This is probably caused by the wide moisture content range, which evidently increases the relative error. But this might also be due to the fact there is always some kind of moisture content distribution in a wood piece that is bigger than the inside of one chip, because of smaller dimensions. The magnetic field is not always quite even, resulting in a small inaccuracy connected with inhomogeneous sample moisture content distribution.

In addition, MR accuracy was also studied with agro biomass samples. Figure 22 shows the results.

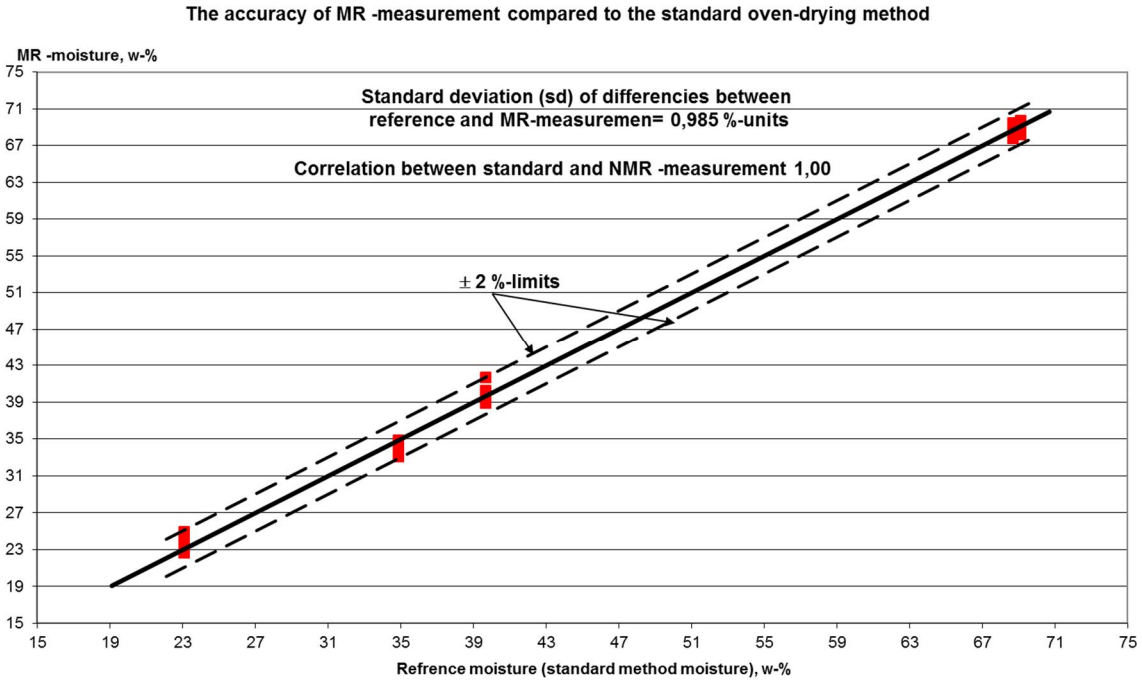

Figure 22. The accuracy of MR measurement compared to the standard oven-drying method with Reed canary grass.

Reed canary grass measurements were carried out directly from samples taken from bales (Fig. 23).

Figure 23. Reed canary grass bale sampling and MR analysis sample.

The grass was not sized but packed directly into the MR sample pot. This meant that it was not easy to get enough grass mass in a pot, especially when the sample was dry. Accordingly, the absolute water quantity remained low, which always weakens the relative accuracy of any instrumental moisture measuring device. Nevertheless, in this case (Fig. 22), uniformity with standard moisture results was still good.

10. Influence of sample dividing (preparation) on accuracy

The increment in sample volume depends on the particle size according to Equation 3 (Solid biofuels. Sampling EN 14778).

$Vol_{incr} = 0.5$, for $d_{95} < 10$ mm
$Vol_{incr} = 0.05 \cdot d_{95}$, for $d_{95} \geq 10$ mm

Equation 3. Calculation of the size of the increment

Vol_{incr} = the minimum volume of the increment, litre
d_{95} = the nominal top size, mm

According to Equation 3, the sample size is 5 litres when the particle size is 100 mm, and 3 litres when the particle size is 60 mm. During the testing of the MR device, the sample size was 5 litres, equalling the 100 mm nominal top size of the material for the sampling subject (Fig. 24).

Figure 24. Sampling from wood chip heaps at a power plant.

MR moisture content gauging was also applied to a five-litre sample so that the sample was carefully divided into two samples: one for standard moisture determination equalling approximately 300 g, and a second one for MR moisture gaug-

ing of approximately 2 x 150 g. In this way, some idea could be achieved of the sample's inherent moisture content deviation's influence on the MR and standard moisture determination. Figures 25 and 26 show the accuracy in the case when sample (5 litres) preparation (dividing) is also taken into account. These studies were made with whole tree chips and Reed canary grass.

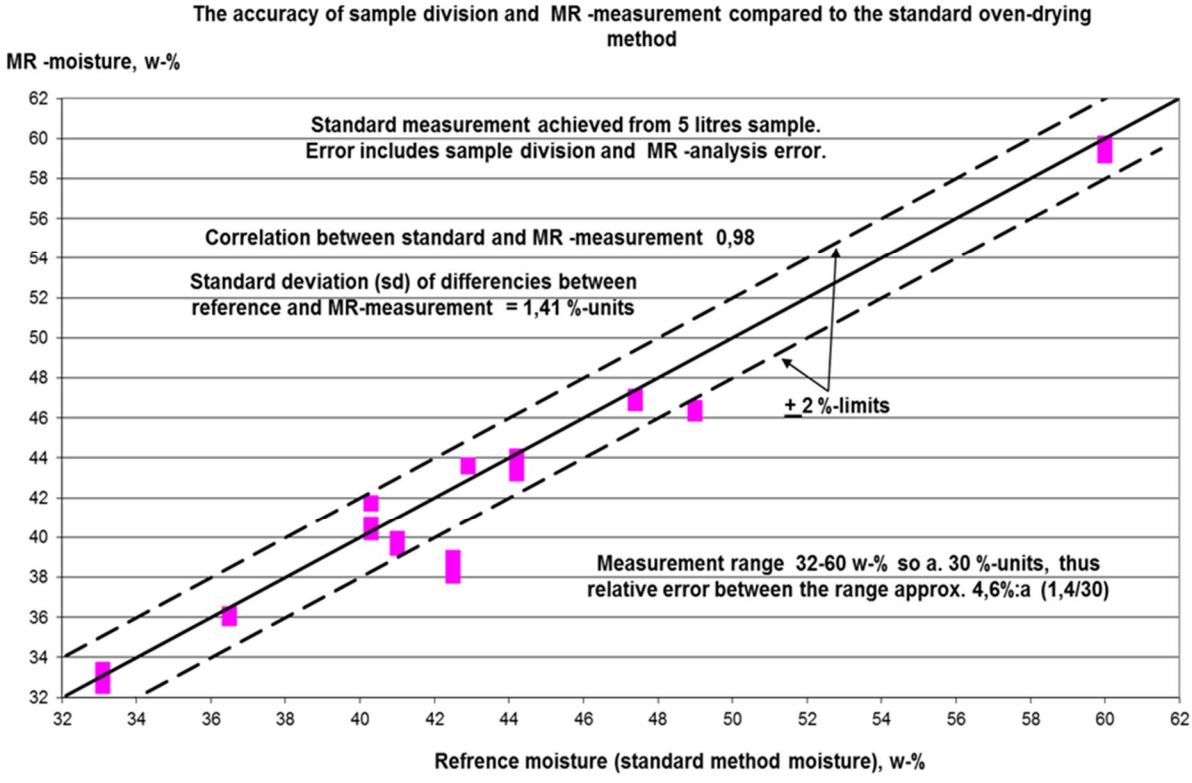

Figure 25. The accuracy of sample division and MR measurement compared to the standard oven-drying method with whole tree chips (including stem and branches).

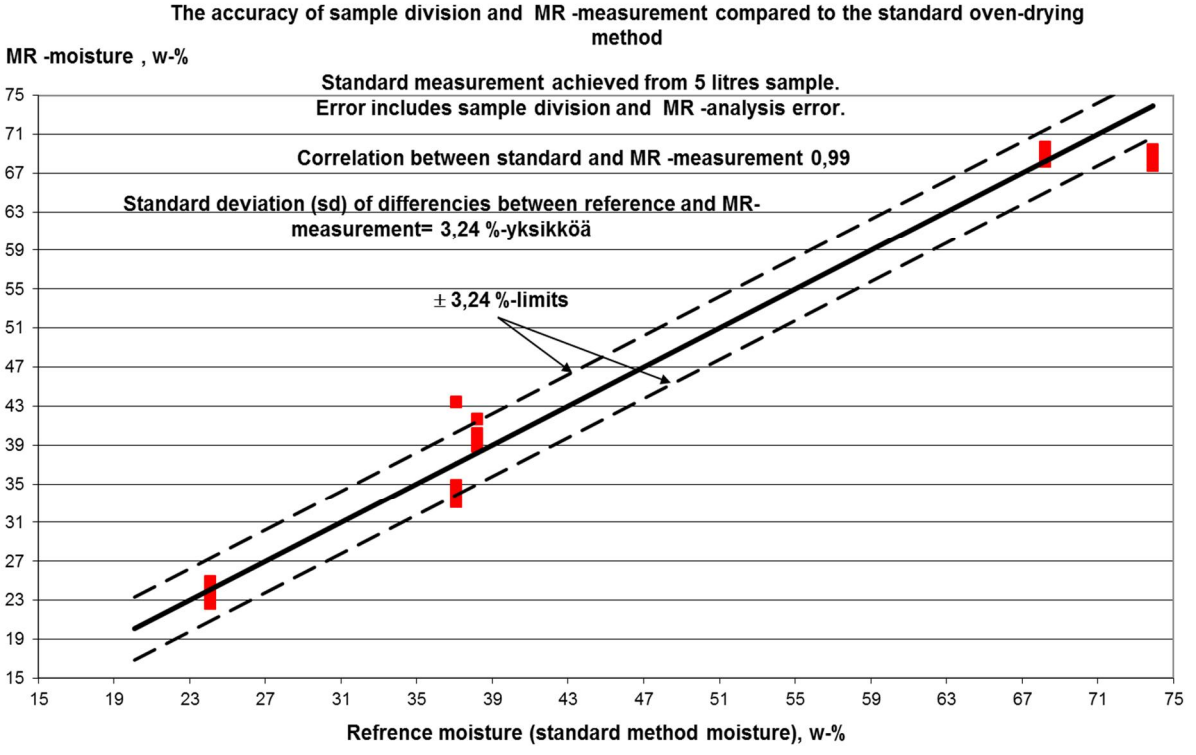

Figure 26. The accuracy of sample division and MR measurement compared to the standard oven-drying method with Reed canary grass.

Figures 25 and 26 show clearly how much the sample preparation, in the case of sample division, affects the accuracy with materials whose inherent moisture content deviation is large. Differences between wood chips and Reed canary grass can also be seen: apparently, the moisture content deviation of the agro biomass is bigger than the wood chip measured. The sample moisture range difference also supports this assumption.

Sample preparation influences the accuracy with biofuels. However, the biggest influences on achieving a representative sample is sampling itself, especially with biofuels acquired from forests, but also with other biomasses. This takes place due to the large moisture content deviation caused by seasonal, storage, and delivery practices and geographical variation, especially in northern conditions. To improve the quality control of biofuels, new standards such as for solid biofuels have been implemented at EU level. Sampling and sample preparation standards (EN 14778 and 14780) and the application of these standards have been continued at national level in Finnish ambient conditions.

11. Utilisation of MR gauging for biofuel moisture content measurement at VTT

When new solid biofuel sampling and sample preparation standards were implemented in Finland, it was revealed quite soon that utilising standard recommended deviation, variance, and precision values would lead to enormous increment sample numbers (ref. Subsection 2.1.1). In particular, this would concern wood fuels from forests whose share of the total energy consumption has increased rapidly and is expected to grow fast in the future. The national target only for forest chips in heat and power generation is 13.5 million solid cubic metres (97 PJ) per annum by the year 2020. Compared, for example, to the year 2007, when forest chips utilised 22 PJ, the increase would be 341% (Heinimö et al. 2011). These figures do not include the possible forest raw material utilisation in second-generation liquid biofuel production. Therefore, a project was set up, with the name CEN adaptation, with energy and forest industry complemented by a few single energy and equipment manufacturing companies. The most important aim of the project was to determine representative moisture and ash content deviations and variance of wood fuels from forests in the conditions predominant in Finland.

The fuels chosen for sampling at separate power plants (six altogether) were forest residue chips, whole tree chips from young cuttings, crushed and chipped stump material, and stem wood chips including bark. The power plants were situated in different parts of the country, form north to south and east to west. It was also very important to carry out sampling during different seasons (winter, summer, and autumn). At four plants, sampling was based on manual sampling (Fig. 27), and two units have mechanical automated sampling equipment that was compared to the manual sampling (Fig. 28). VTT was the organisation responsible for performing the project, and manual sampling was executed obeying the instructions of the new sampling standard (EN 14778).

Figure 27. Manual sampling from forest residues. Left: start of container discharge, and right: sampling made by truck driver and by project personnel. Segregation can be noticed in the load (left picture)!

Figure 28. Mechanical sampling (left) and manual sampling from the same load (right).

From the very beginning of the project, it was realised that the sample and determination number would be huge. The aim was to take 10 increment samples from each truck load or equal quantity of each fuel type. Several trucks delivering certain fuel types were subjected to daily sampling. In addition, it was planned to determine moisture content from each increment sample to collect the data to calculate fuel moisture content deviation in a load. Ash determinations were limited to stump material because there was a shortage of data available concerning the stump chips. All the figures were averaged in a table, which can be used as a basis for further evaluations and calculations. The sampling precision suitable in northern conditions for forest fuels was settled. The project produced a lot of additional information about wood fuel quality and its changes during the seasons, as well as sampling accuracy at different power plants. Hints for improvement and

application of sampling standards were delivered. In addition, two “main” mechanical sampling equipment principles were tested and compared to manual sampling, according to the new sampling standard.

Because of the increased sample quantities following a vast number of moisture measurements, it was decided to utilise a rapid instrumental moisture gauging device instead of standard moisture content determination based on oven-drying. The purpose was to save time and labour, because the planned project schedule forced a strict budget and timetable. At the same time, Vaisala Ltd was developing moisture content measurement based on MR technology. VTT was also involved in the development work in the sense of prototype testing. VTT studied the accuracy of the MR device on different biofuels from forests and fields. These tests demonstrated that metering by the MR principle is a very accurate and reliable method of moisture content determination compared to the other alternatives. In addition, its calibration is easy and rapid, using only pure water containing a small quantity of ferrous salts. Material-specific calibration curves are not needed. Vaisala provided VTT with an MR moisture prototype gauge for use in the CEN adaptation project`s moisture measurements.

The first effort at instrumental moisture gauging was to ensure the adequate basic accuracy described in section 9. To achieve a similarity with the standard (EN 14774) method, two MR measurements were always made from one increment sample. Weighted average values were used in calculations. In all cases, one 5-litre increment sample was carefully divided into two MR analysis samples, with approximately 150–200 g sample material in each pot (Fig. 29). One load’s analysis included 20 single MR measurements. Before the division, the increment was always shaken in a 10-litre bucket to be properly blended. The procedure was similar to that used in basic accuracy testing.

Figure 29. Increment sample division (left) into two MR analysis samples and one truck load’s increments (10) in MR analysis pots (right).

The second effort when using the instrumental method is to ensure that the gauge is reliable in the longer term. Therefore, the MR device was calibrated every day

before the measurements and often during the work if something unexpected happened, such as a disturbance in the ambient conditions, like a drastic temperature change (hall doors were opened during winter). After every calibration, a reference was made by measuring the biomass samples whose moisture content was known (Fig. 30). These samples were also measured after each 10-sample measurement period. Figure 30 represents the measurement of known samples over a two-month period in 2011. It shows that the MR measurement precision has stayed at a high level.

The third task was to determine the preparation and testing variance (V_{PT}) when using an MR device following sampling standard (EN 14778) instructions (ref. Equation 1). To improve the V_{PT} value, it was decided to make two separate MR measurements from each 5-litre increment sample (ref. section 10).

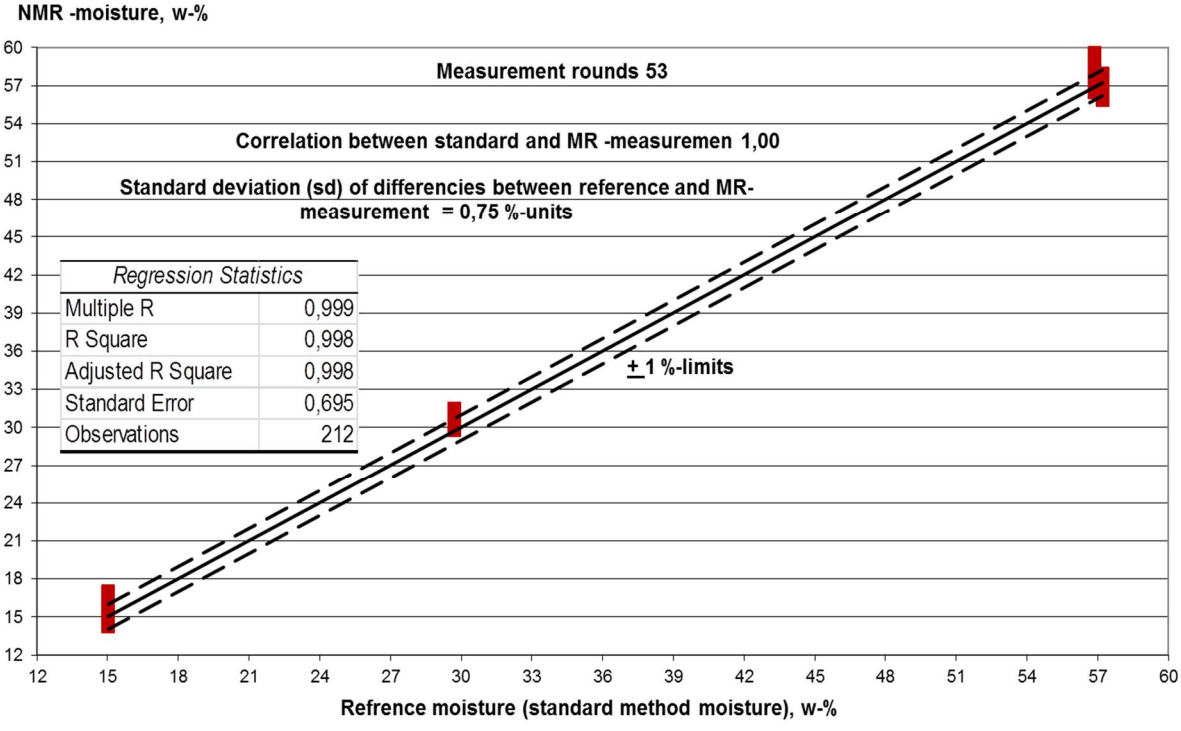

Figure 30. The MR accuracy reference by measuring samples with a certain known moisture. content.

12. Usability, benefits, and limitations of MR devices according to the VTT experience

The MR measurement principle has been known and utilised already since the early 1950s. It has become the basic instrumental analysis tool in chemistry. It is also well known as a very accurate method of analysing most compounds, especially substances containing hydrogen. Nowadays, MR devices are utilised mostly in spectroscopy and structural analysis. The utilisation of MR metering has expanded extensively to medical diagnostics as a form of magnetic resonance imaging (MRI). Because of the precision of the MR principle, efforts have been made for a long time to apply it in new different areas and make it more user friendly, smaller, and even portable. Mostly, the challenge has been how to control magnet size and achieve an even magnetic field using smaller equipment. Modern instrumental technology has provided the chance to enhance magnets and RF-sending and receiving facilities, and, first of all, signal processing and analysing possibilities. Therefore, new efforts have been made to create simpler and even portable MR gauges, simply for moisture content gauging of different substances such as biofuel.

According to our experience, the MR principle is also an accurate method of measuring biofuel moisture content. As matter of fact, it has been the most precise gauge compared to the analogous method utilised in the same way in “at line” measurements for biofuels in VTT. That is why the MR device was selected to be used in a project for validation and adaption of a new solid biofuel sampling standard (EN 14778) for the conditions prevailing in Finland. The “absolute” precision could be confirmed because the MR measurement does not change the sample characteristics under metering, allowing verification of the reference moisture content according to the standard (EN 14774). The same system was utilised for all measurement types (gauges), giving each one the same chance. The decision on the selection of the measurement method was made according to those results.

A very significant issue when using an MR device is the simple calibration procedure of the meter. The calibration takes place using only pure water with some added metal salt, as mentioned previously. It is also essential that calibration is valid for many kinds of materials in addition to biofuel mixtures. This means that no material-specific calibration curves are necessary. This is a substantial benefit

compared to most gauges utilised for the same purpose. The MR device also turned out to be reliable in the long term, giving reproducible results. One reason for that is, of course, the simple calibration procedure.

Most limits to utilising MR gauging are connected with temperature sensitivity and the measurement of minimal absolute quantities of water in a sample, which are common challenges to all instrumental methods. It is important in the calibration of an MR device to carry it out at the same temperature as the measurement takes place. Ambient temperature changes should also be avoided during measurements In particular, Vaisala`s MR prototype was sensitive to cold or hot air streams on the exposed magnet. The prototype's magnet was not covered with any shelter, but was quite open, with only the surface painted with varnish (Fig. 31).

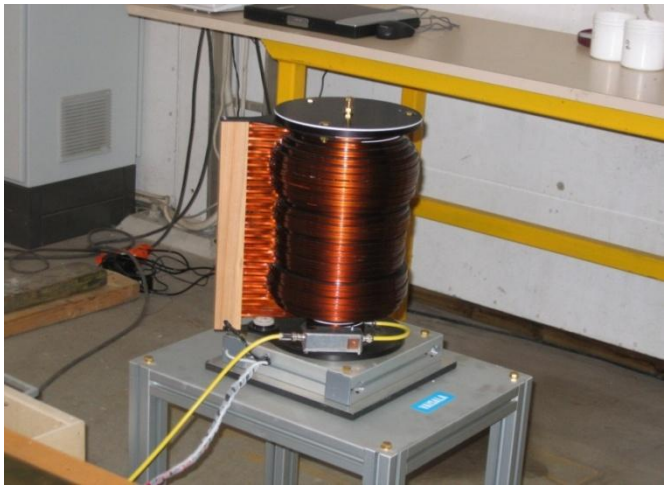

Figure 31. Vaisala`s MR prototype's electromagnet.

Testing the MR measurement accuracy in a case where the sample contains a small absolute quantity of water can be seen in Figure 32. The material in this case has been different torrefied pellet samples. Figure 32 illustrates how there can be an error in MR measurements that probably partly seems to be systematic. A systematic error can be reduced by signal processing, but in this case cannot be totally removed.

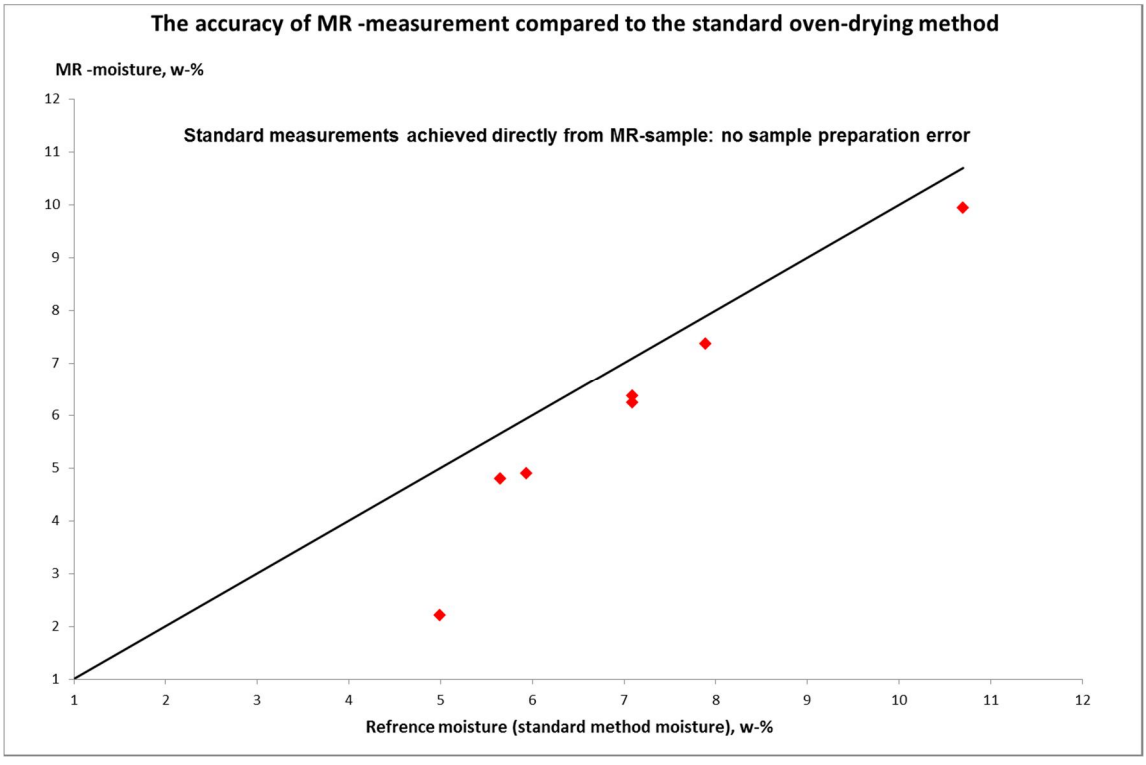

Figure 32. The accuracy of MR measurement compared to the standard oven-drying method with pellets made from different torrefied raw materials.

Vaisala also took the error account described above into their instructions. It was recommended to avoid measurement in a case where a sample contains less than 20 g H_2O. This moisture (water) content means approximately 10 w-% relative moisture content of an MR sample. In rare cases, biofuel moisture content is even under 20 w-%. Normally, the biofuel moisture range begins from 20 w-% and is between 20 and 65 w-%.

An MR device is sensitive to ambient electric and magnetic fields and big ferromagnetic metal structures located near the place where the measurements are taken. At VTT, the MR gauge was sited in a hall reserved for quite large experiments. There are lots of steel constructions and also electrified equipment and wires. Vaisala had defined the minimum distance (60 cm) from objects that may cause disturbance in MR measurement. The instructions tried to obey as carefully as possible, but couldn`t totally followed. Nevertheless, no difficulties caused by steel constructions or electrical fields were noticed. Instead, it was found that there was some sensitivity of the weighing element (strain gage) to vibration caused by, for example, a fork-lift truck passing by.

Maybe the most important deficiency of Vaisala`s MR prototype in the moisture content definition was a slightly inadequate volume for the MR sample pot (630 ml). Therefore, in VTT`s actions, a replication sample was always measured as one increment sample, as the standard method demands. The standard method (EN 14774) presupposes at least a 300 g sample mass dried in an oven, but no replication is needed. From the actual measurement point of view, it is not very decisive, because MR gauging is very fast and the results are available in a few seconds. But due to the sample preparation, more labour is needed and surplus sample division increases the possibility of error.

13. Further development of MR moisture measurement technology

In all industrial processes, control systems need continuous and reliable information from the processes to act properly. This means extended online measurements applying to meter the most important characteristics of the feedstock involved. The development of online measurement is significantly challenging compared to sampling based on “at-line” principle gauging systems. One good example of that is the online moisture content measurement of biomass fuels. There is no reliable online moisture gauging method available on the market at the moment. Near infrared absorption, utilising the whole spectral measurement can be applied to homogenous materials (e.g. paper) in which moisture is evenly distributed. It has still turned out to be challenging to create a global calibration model for inhomogeneous substances. Microwave-based methods are also still under development for online purposes. The difficulties cause material-specific calibration and extensive, transient density variations. In principle, the MR measuring method would not be affected by the circumstances and substance variations described for other alternative metering principles. The main challenge in developing direct MR gauging for online applications is the large installation, leading to high investment costs. This takes place basically due to the effective magnet needed for “conveyor or silo” measurements. This trouble can be avoided by developing mechanical and automated sampling systems. This procedure was already, to some extent, studied by Vaisala (Fig. 33).

Figure 33. MR measurement testing as an online application at VTT`s PDU line.

It was rapidly discovered that representative sampling is very difficult when utilising such a small sampling pot (small sample size) as a part of a mechanical sampler device. Therefore, it was discussed whether to develop MR measurement further by increasing the sample pot size. The target would be the pot volume, enabling measurement at once of the biofuel sample mass, which is in accordance with the moisture content determination standard (EN 14774) demands of 300 g. The quantity (mass) would presuppose an approximate volume of one litre. Only small changes would have been necessary concerning the structure of Vaisala`s MR prototype. The size of the magnet would remain unchanged, which would mostly influence the usability and portability. The statistics and changes affecting precision have to be studied after the revision.

Generally reviewed, the enlargement of a sample pot also makes “at-line” MR moisture gauging more comparable with standard measurements. It reduces labour in sample handling, and sample preparation errors will also decrease. Bigger particles of a biofuel sample can also be measured without sizing. The actual size of a new pot is designed to be nearly twice as big as the pots presented in Figure 34.

Figure 34. Two MR-prototype sample pots containing crushed wood fuel material.

The MR measurement principle could also be utilised for measuring on-line at conveyors. Most easily, the system could be assembled around pipes or tubular conveyors (tube conveyors). This kind of facility would need large and effective magnetic fields. It has also to take into account that steel (metal) construction and steel enforcement of a belt are not possible in this case. The facilities would be like MRI applications in medical diagnostics. The benefits would be the possibility to achieve a lot of other kinds of information than only moisture content from the material to be measured on a conveyor. At the moment, on-line facility investment in the manner described would still be so heavy that its application for biofuel measurement does not seem to be feasible. The material subjected to the measurement should be more valuable. Maybe the first online MR applications will be seen in the manufacture of medicines.

14. Conclusions

MR moisture content measurement gives the opportunity to determine rapidly and accurately different biofuels` moisture content, according to the experience achieved by utilising the prototype gauge at VTT for one year in 2011. The absolute precision seems to be comparable to the standard moisture content measuring method (EN 14774). The gauge was reliable and easily calibrated. For the calibration, only water containing some common metal salt is needed. Material-specific calibration curves are not necessary.

The prototype was based on the “at-line” principle, which means that representative sampling is necessary for instrumental metering. MR gauging is sensitive to temperature, and calibration should be carried out at same temperature as the actual measurements. Frozen samples cannot be measured. Sensitivity to ambient electrical fields and metal constructions was less of a problem than expected. The prototype device was affected by ambient vibrations, influencing the strain gage of the sample weighing system.

The biggest challenge in using the MR prototype gauge is caused by the sample pot volume, which was a little more than half a litre (630 ml). The maximum mass of some biofuel samples reached approximately 200 g, and on average about 150 g. This is half of the mass presupposed by standard EN 14774 to use in oven-drying. Therefore, at VTT, two separate samples were applied in MR gauging and the average result was utilised in comparisons and calculations. Small sample volumes also increase sample preparation errors. From the very beginning, the prototype supplier (Vaisala Ltd) realised it was necessary to increase sample quantity. Therefore, sample pots were tested that were almost twice as big as the normally used pots, without making any extensive changes to the structure of the prototype MR gauge. It is important to be able to keep the size of the magnet sufficiently small, which makes the device usable and portable. The test results were not available for VTT.

VTT also participated in the further development efforts carried out by Vaisala. It was clearly understood that to develop MR measurement as a continuous-principle workable meter, it should somehow be combined with a representative mechanical sampling system. An MR system acting directly on the conveyor line would need heavy constructions, leading to high investment costs. In that case, the system would be similar to the MRI equipment utilised in medical diagnostics.

In tests at VTT, it turned out that according to the online principle, the acting prototype device also had too small a sample pot, decreasing the accuracy because of unrepresentative sampling. The experiments also emphasised the importance of increasing the size of the sample pot as a next step in developing a precise and reliable instrumental device for biofuel moisture content measurement.

15. Summary

Biomass is extensively utilised in energy production and as a raw material, such as in the production of liquid biofuels. All those processes will benefit if the moisture content of bio material is known in advance as accurately as possible under transient circumstances. Biofuel trade is extensively based on the calorific value of fuels. As a first step, this also increases the need for rapid and accurate moisture content determination. During the last few years, much biofuel standardisation has been implemented, emphasising biofuel quality control at all stages of the utilisation chain.

In principle, moisture instrumental measuring can utilise many technologies and procedures. Typical techniques are infrared (ir, nir), radiofrequency (rf), microwave, radiometric, electrical conductivity, capacitance, and impedance. Nuclear magnetic resonance (nmr) and thermal neutron absorption are also applied.

The MR measurement principle has been known and utilised already since the early 1950s. It has become the basic instrumental analysis tool in chemistry. It is also well known as a very accurate method of analysing most compounds, especially substances containing hydrogen. Nowadays, MR devices are utilised mostly in spectroscopy and structural analysis. The utilisation of MR metering has expanded extensively to medical diagnostics as a form of magnetic resonance imaging (MRI). Because of the precision of the MR principle, efforts have already been made for a long time to apply it in new different areas and to make more user friendly, smaller, and even portable devices. Mostly, the challenge has been how to control the magnet size and achieve an even magnetic field using smaller equipment. Modern instrumental technology has provided the chance to enhance the magnets and RF-sending and -receiving facilities, and, first of all, the signal processing and analysing possibilities. Therefore, new efforts have been made to create simpler and even portable MR gauges also simply for moisture content gauging of different substances, such as biofuel.

VTT has utilised Vaisala`s MR prototype for approximately one year (in 2011), for moisture content measurement of different biofuels. The first step in the use of the MR device for moisture determination was the definition of its measurement accuracy compared to the standard method (EN 14774). Accuracy testing of the MR device was carried out by first measuring the sample moisture content using an MR gauge, and immediately after that setting the same sample in an oven

according to the standard (EN 14774). The testing method is possible because the sample characteristics, especially concerning moisture content, do not change during MR measurement, which takes a few seconds. Those tests proved that the absolute precision seems to be comparable to the standard moisture content measuring method. The gauge was reliable and easily calibrated. For the calibration, only water containing some common metal salt was needed. Material-specific calibration curves are not necessary. As matter of fact, it has been the most precise gauge compared to the analogous method utilised in the same way for “at-line” measurements of biofuels at VTT.

The biggest challenge in using the MR prototype gauge was caused by the sample pot volume, which was 630 ml. The average mass of the biofuel samples reached approximately 150 g. This is half of the mass presupposed by standard EN 14774 to use in oven-drying. Therefore, at VTT, two separate samples were applied in MR gauging and the average result was utilised in comparisons and calculations. Small sample volumes also increase sample preparation errors. From the very beginning, it was planned to increase the sample volume. Vaisala also tested bigger sample pots in the prototype without any substantial structural change.

VTT also participated in the further MR gauging development efforts carried out by Vaisala. It was clearly understood that, to develop MR measurement as a continuous-principle workable meter, it should somehow be combined with a representative mechanical sampling system. An MR system directly acting on the conveyor line would need heavy construction, leading to high investment costs. In that case, the system would be similar to the MRI equipment utilised in medical diagnostics.

In tests at VTT, it turned out that, according to the online principle, the acting prototype device also had too small a sample pot, decreasing the accuracy because of unrepresentative sampling. The experiments also emphasised the importance of enlarging the sample pot as the next step in developing a precise and reliable instrumental device for biofuel moisture content measurement.

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Title	**Rapid and accurate biofuel moisture content gauging using magnetic resonance measurement technology**
Author(s)	Timo Järvinen
Abstract	Biomass is extensively utilised in energy production and as a raw material, such as for the production of liquid biofuels. All those processes will benefit if the moisture content of bio material is known in advance as accurately as possible under transient circumstances. Biofuel trade is increasingly based on the calorific value of fuels. In the first step, this also increases the need for rapid and accurate moisture content determination. During the last few years, large biofuel standardisation has been implemented, emphasising biofuel quality control at all stages of the utilisation chain. In principle, the moisture instrumental measurement can be utilised by many technologies and procedures. Typical techniques are infrared, radiofrequency, micro-wave, radiometric, electrical conductivity, capacitance, and impedance. Nuclear magnetic resonance (MR) and thermal neutron absorption are also applied. The MR measurement principle has been known and utilised already since the early 1950s. It has become the basic instrumental analysis tool in chemistry. It is also well-known as a very accurate method for analysing most compounds, especially substances containing hydrogen. The utilisation of MR metering is expanded extensively to medical diagnostics as a form of magnetic resonance imaging (MRI). Because of the precision of the MR principle, there have for a long time been efforts to apply it in new and different areas, and to make more user-friendly, smaller, and even portable devices. Such a device was designed by Vaisala a few years ago. VTT has utilised Vaisala`s MR prototype for approximately one year for moisture content measurement of different biofuels. The first step in the use of an MR device for moisture determination was the definition of its measurement accuracy compared to the standard method (EN 14774). Those tests proved that the absolute precision seems to be comparable to the standard moisture content measurement method. It was also found out that the MR gauge was the most precise device utilised in the same way, when compared to other alternatives. The gauge was also reliable and easily calibrated. The biggest challenge in using the MR prototype gauge was caused by the volume of sample pots. The average mass of biofuel samples reached about half of the mass presupposed by standard EN 14774 for oven drying. Therefore, at VTT, two separate parallel samples were applied for MR gauging, and the average result was utilised in comparisons and calculations. Already, Vaisala tested the prototype, applying approximately a sample pot twice as big as that used in the prototype, and Metso Automation has recently realised this improvement.
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Julkaisun sarja ja numero
VTT Technology 89

Nimeke	**Nopea ja tarkka biopolttoaineiden kosteuden määritys käyttäen magneettisen resonanssin mittaukseen perustuvaa laitetta**
Tekijä(t)	Timo Järvinen
Tiivistelmä	Biomassaa käytetään paljon polttoaineena, ja sen käyttö kasvaa energialähteenä sekä raaka-aineena nestemäisten biopolttoaineiden valmistuksessa. Kaikki nämä prosessit hyötyvät, jos biomassan kosteus tunnetaan etukäteen kaikissa oloissa. Biopolttoaineiden kauppa perustuu yhä enemmän polttoaineen energiasisältöön, joka lisää nopean ja tarkan kosteuspitoisuuden määrityksen tarvetta. Viime vuosina on EU-tasolla toteutettu myös laaja biopolttoaineiden standardointi, joka korostaa laadunhallinnan ja laatutietojen tärkeyttä hankinta- ja toimitusketjussa. Periaatteessa kosteuspitoisuutta voidaan mitata monella instrumentaalimenetelmällä. Tyypillisiä tekniikoita ovat mm. infrapuna (ir, nir), radiotaajuus (rf), mikroaalto, radiometriset, sähkönjohtavuuteen ja kapasitanssiin perustuvat laitteet. Myös magneettista resonanssia (MR) ja termisten neutronien absorptiota on käytetty. MR-menetelmän periaatteet on tunnettu ja menetelmää on käytetty jo 1950-luvulta lähtien. Siitä on tullut paljon käytetty instrumentaalianalyysimenetelmä kemiassa. Se on myös tunnettu tarkkana menetelmänä, jota soveltuu erilaisten yhdisteiden analyysiin ja erityisesti vetyä sisältävien aineiden tutkimiseen. Nykyisin MR-tekniikkaa käytetään spektroskopiassa ja rakenneanalyysissa. MR-teknologian hyödyntäminen on laajentunut lääketieteelliseen diagnostiikkaan magneettikuvauksena (MRI). Jo kauan on pyritty kehittämään pienempiä MR-mittareita. Vaisala Oyj toteutti muutama vuosi sitten tällaisen laitteen. VTT on käyttänyt Vaisala Oyj:n kehittämää MR-prototyyppilaitetta noin vuoden ajan vuonna 2011 eri biopolttoaineiden kosteuden mittauksessa. Ensimmäinen vaihe VTT:llä oli määrittää laitteen tarkkuus kosteusmittauksessa verrattuna standardissa (SFS-EN 14774) kuvattuun uunikuivausmenetelmään. Nämä testit osoittivat, että prototyypin tarkkuus oli verrattavissa standardin mukaiseen kosteuspitoisuuden määritykseen. MR-mittaus oli myös tarkempi kuin muut vastaavantyyppiset samalla tavalla käytettävät instrumentaalilaitteet, joita VTT:llä oli ollut käytössä. Prototyyppi oli lisäksi luotettava ja helposti kalibroitavissa. Suurin haaste MR-prototyypin käytössä oli mittausastian koko. Siksi astiaan pystyttiin panemaan tyypillistä biopolttoainetta keskimäärin noin puolet siitä, mitä uunikuivaukseen perustuvassa näytteenottostandardissa (SFS-EN 14774) edellytetään näytemassaksi. Sen vuoksi VTT:n mittauksissa kosteus määritettiin MR-laitteella aina kahdesta samasta näytteestä otetusta osanäytteestä. Laskelmissa ja vertailuissa käytettiin kahden mittauksen keskiarvoa. Jo Vaisalassa prototyypillä testattiin kaksi kertaa suurempaa näyteastiaa, jonka Metso Automaatio on ottanut käyttöön kaupallisissa laitteissa.
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