Measuring Forest Fuel Quality for Trade and Production Management

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Abstract

An increased use of forest fuels resulted in a new Timber Measurement Act, specifying the requirements for measurements of these assortments. The law has increased the demands when measuring quality parameters, e.g. moisture and ash content. This thesis aims to a) develop a robust validation method for measurement precision and accuracy; b) validate instruments using electric capacitance (CAP), magnetic resonance (MR), near infrared spectroscopy (NIR), and X-ray technologies for moisture content determination; and c) evaluate the possibility to determine other parameters using x-ray data. The tested instruments had similar measurement precision. All except for the CAP produced 95% of their measurements within less than ± 2.5 percentage points of the mean. The accuracy of moisture content measurements varied between; instruments, moisture content classes, and forest fuel materials, as well as between frozen and unfrozen materials. MR was the only instrument that was not sensitive to fuel material. MR showed the highest measuring accuracy and CAP the lowest. The X-ray instrument could determine ash content and net calorific value, but the latter needed calibration. It was possible to estimate the proportion of fines and median particle size accurately using X-ray data. The capacitance instrument is easy to transport and use in the field, but the other instruments are intended for use at a measurement station.

A calibration of the instruments would benefit from precisely defined fuel assortments, as would the customers, who could use it to optimize combustion. Automated methods to verify the delivered fuel assortment are required, e.g. by NIR or X-ray sensors. The oven-drying method and all studied instruments determine moisture content on samples, and the sampling procedure is crucial to accurately estimate the mean and sample variance. The sampling problem could be minimised if measurements could involve most of the load otherwise a sufficient number of samples must be taken. A cost-efficient measurement procedure requires a balance of sampling intensity vs measurement costs and benefits. A fast moisture measurement procedure that enables customer to optimize combustion can be more cost-effective than the oven-drying method, especially if it provides additional data.

Keywords: moisture content, ash content, net calorific value, energy, chips, logging residue, bioenergy

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Mätning av skogsbränslets kvalité för handel och produktionsstyrning

Sammanfattning

Användningen av primära skogsbränslen ökade snabbt under tjugohundratalets första decennium. Detta ledde till en virkesmätningslag med ökade krav på noggrannhet och precision. Målet för denna avhandling var a) att utveckla en statistiskt robust valideringsmetod för precision och noggrannhet vid fukthaltsmätning, b) att validera mätinstrument som använder elektrisk kapacitans (CAP), magnetresonans (MR), nära-infraröd spektroskopi (NIR) eller röntgenteknik för fukthaltsmätning i flis och c) att utvärdera möjligheterna att skatta andra parametrar med hjälp av röntgenteknik. Resultaten visade att, frånsett CAP mätaren, hade instrumenten en precision där 95% av mätningarna återfanns inom ± 2,5%-enheter. Det fanns stora variationer i noggrannhet för fukthalt som påverkades av: instrumenttyp, fukthaltsklass på materialet, typen av material, och om materialet var fruset eller ej. MR-instrumentet uppvisade den högsta och CAP den lägsta noggrannheten men MR var enda instrument okänsligt för material. Röntgeninstrumentet kunde även skatta askhalt och effektivt värmevärde, och med möjlighet att skatta både finfraktion och medianpartikelstorlek med förhållandevis stor säkerhet. Endast CAP instrumentet är lätt att transportera och använda i fält, övriga instrument är avsedda att användas på mätstationer vid större terminaler eller mottagningsplatser.

En sortimentsbeskrivning av flisen skulle underlätta kalibreringen av instrumenten, och dessutom gagna bränslekunderna för att öka effektiviteten i sina anläggningar. Men för detta måste det gå att verifiera att levererat bränsle överensstämmer med sortimentet, vilket skulle kunna göras med sensorbaserade instrument. Såväl ugnsmetoden som instrumenten fastställer fukthalten för prover, vilket kräver korrekt sampling för en korrekt fukthaltsbestämning. Provtagningsproblemet kan minimeras om mätningar kan utföras för större delen av leveransen annars måste ett tillräckligt stort antal prover tas. Ett kostnadseffektivt mätförfarande kräver en balans mellan provtagningsintensiteten och mätningens kostnader och fördelar. En snabb mätning av fuktmätning som gör det möjligt för kunden att optimera förbränningen kan vara mer kostnadseffektiv än ugnstorkningsmetoden, speciellt om det ger ytterligare egenskapsdata

Nyckelord: fukthalt, askhalt, värmevärde, energi, flis, grot, bioenergi

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Dedication

To my family.

Contents

List of publications

This thesis is based on the work contained in the following papers, referred to by Roman numerals in the text:

I Lars Fridh*, Lars Eliasson & Dan Bergström (2017). Precision in moisture content determination of wood fuel chips using a handheld electric moisture meter. *Submitted*

II Lars Fridh, Sylvain Volpé & Lars Eliasson* (2014). An accurate and fast method for moisture content determination. *International Journal of Forest Engineering* 25:3, 222-228, DOI: 10.1080/14942119.2014.974882.

III Lars Fridh*, Sylvain Volpé & Lars Eliasson (2017). A NIR machine for moisture content measurements of forest biomass in frozen and unfrozen conditions. *International Journal of Forest Engineering* 28:1, 42-46, DOI: 10.1080/14942119.2017.1297521

IV Lars Fridh*, Sylvia Larsson, Dan Bergström, Lars Eliasson & Tomas Thierfelder (2017). Combining two X-ray methods for simultaneous determination of net calorific value, moisture- and ash content in biomass chips. (*Manuscript*)

V Lars Fridh*, Sylvia Larsson, Dan Bergström, Lars Eliasson & Tomas Thierfelder (2017). Combining two X-ray methods for determination of particle size and fines of forest biomass chips. *(Manuscript)*

Papers II-III are reproduced with the permission of the publishers.

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The contribution of Lars Fridh to the papers included in this thesis was as follows:

I Planned the study design. Conducted data collection with co-writers. Conducted all analysis. Wrote the manuscript with input from co-writers.

II Planned the study design, and conducted data collection with co-writers. Conducted the analysis. Wrote the manuscript with co-writers.

III Planned the study design, and conducted data collection with co-writers. Conducted the analysis. Wrote the manuscript with input from co-writers.

IV Planned the study design, and supervised the data collection. Conducted the analysis. Wrote the manuscript with input from co-writers.

V Planned the study design, and supervised the data collection. Worked with co-authors on data analysis and preparing the manuscript.

Abbreviations

A	Ash content, % dry matter
AC	Alternating current
CAP	Electric capacitance
CLA	Cluster analysis
CXR	Combined DXA and XRF technology
DXA	Dual-energy X-ray absorptiometry
F	Fines, particles < 3.15 mm
GRM	General regression model
M	Moisture content, % wet basis
M_CL	Moisture content class
M_CON	Moisture condition, frozen or non-frozen
MDS	Multidimensional scaling
MR	Nuclear magnetic resonance
NIR	Near-infrared spectroscopy
P	Particle size class,
PCA	Principal component analysis
PLS	Partial least squares
pp	Percentage point, arithmetic difference between two percentage values
q	Net calorific value (as received)
SORT	Forest biomass fuel material (assortment)
XR	X-ray
XRF	X-ray fluorescence spectroscopy

1 Introduction

1.1 Forest fuel supply for heat and power generation

Heat and power generation using forest fuel is well-developed in Sweden and comprises much of the country's energy infrastructure. Approximately 90% of apartment buildings are heated by more than 500 district heating networks (SVEBIO, 2016b). Of these, 209 are combined heat and power plants (CHP) also producing electricity (SVEBIO, 2016a). In 2015, the wood fuel-based production at CHPs was 51.4 TWh of heat and 3.7 TWh of electricity, and at heating plants 8.6 TWh of heat (SCB, 2016). Of the wood fuel, 15.3 TWh was supplied as primary forest fuel (Anon., 2016a).

Demand is highly dependent on outside temperature (Ericsson & Werner, 2016). The resultant fluctuations place heavy demands on the supply system, with winter biomass supply a particular strain (Gadd & Werner, 2013; Dotzauer, 2002). Consumers of primary forest fuels are mainly large energy companies, forest companies and municipal heating companies, while the supply side consists of both small, private forest owners and large forest companies (Olsson et al., 2016). In the supply chain, the biomass may be traded several times; for example, the forest owner sells the forest fuel assortment together with saw logs and pulpwood to one company that, in turn, sells the forest fuel to a forest fuel supply company that sells it to the end customer.

Primary forest fuels are currently divided into four categories based on the source of the material: (1) energy wood, i.e. defect logs (stem wood) that are unsuitable for saw or pulp and paper mills (4.9 TWh); (2) logging residues, i.e. tops and branches from final felling (9.0 TWh); (3) small trees, i.e. trees from early thinning (1.1 TWh); and (4) stumps (0.2 TWh) (Anon., 2016a). Forest owners, forest companies, forest owners associations, and biofuel companies are all active in the storage, logistics and fuel management before the forest fuel reaches the heating plants. For transparency on the biomass fuel market, the products traded must be well defined and the measurement procedures relating to quality and quantity of the delivered fuel must be both accurate and cost effective.

A heating plant's conversion technology determines its fuel property requirements (Strömberg & Svärd, 2012). Briefly, a specific heating plant solution is designed for a fuel with a given ash content, moisture content and particle size or size distribution (ISO, 2014). These properties vary within a certain range, but if the deviation from stipulated properties is too great, the risk of various operational problems or reduced efficiency increases (Bäfver & Renström, 2013).

Moisture content, because it strongly influences the net calorific value, is generally the most important forest fuel quality parameter (cf. ISO, 2014) but a minimum moisture content is not always the most desirable. For example, the desired moisture content for a district heating plant may vary over time, from >55%, when flue gas condensation is utilised for heat production, to <40% when more electricity is produced. Another important quality parameter is the fuel chip fraction or particle size distribution (PSD). The optimal PSD for a combustion facility varies according to boiler type, fuel handling, storage and mixing, and operating strategy. Oversized material is known to cause stops in the feeding mechanisms and high amounts of fine fractions can generate combustion problems, as it follows the gas stream and burns in the wrong part of the boiler (Johannesson & Njurell, 2014; Bäfver & Renström, 2013). Primary forest fuels have relatively heterogeneous properties as they are made up of different fractions from the trees, such as stem wood, bark, branches and needles. In contrast, forest industrial residues, such as chips, sawdust, and bark, are relatively homogeneous. However, one common requirement is for a consistent fuel quality, particularly in terms of moisture content and particle size distribution.

Forest fuels are stored for a considerable period after harvesting. A recommendation is to store logging residues on the clear-cut over one summer (Skogsstyrelsen, 2008), which gives an average storage time of about a year for the residues. Roundwood logs have better storage properties and are often stored for long periods. It is not uncommon for forest fuels to be stored for up to 18-24 months before delivery to a heating plant.

Storage entails risk for major changes in fuel quality, decomposition and, for comminuted fuels, self-ignition (Olsson et al., 2016; Jirjis, 1995). Approximately 80% of forest fuels are delivered directly to the customer from storage on a roadside landing before delivery to heating plant (Figure 1, systems B & E). The remaining 20% passes through a forest fuel storage terminal

(Björklund, 2014) (Figure 1, systems A, C & D) before delivery to the customers. These storage terminals are often small and lack permanent staff and equipment for moisture content determination (Kons et al., 2014).

Measurement procedures for traded forest fuels are regulated in the Swedish Timber Measurement Act of 2015 (Anon., 2014a; Anon., 2014b). This regulation is limited to the first level of trade, when a forest owner sells the fuel to the first buyer in the supply chain, i.e. when the fuel reaches the market. The legislation requires quantity and quality measurements in the first level of trade to ensure correct payment to the forest owner (Figure 1). Normally, a timber buyer organisation has made an agreement with the forest owner for the saw timber, pulpwood, and forest fuel. The timber buyer then sells the forest fuel to a biofuel company that has a delivery agreement with the heating plant.

Fuel quality is generally measured when the biomass is delivered to the heating plant. Measurements can be carried out in any way agreed by the parties if data is used only to regulate the payments between the timber buyer and the biofuel company and between the biofuel company and the heating plant. If the same measurements are to be used as a basis for payment to the forest owner, provisions in the Timber Measurement Act apply, with specific measurement procedure requirements. This has put forest fuel quality measurements into a whole new perspective, with many new challenges for all parties.

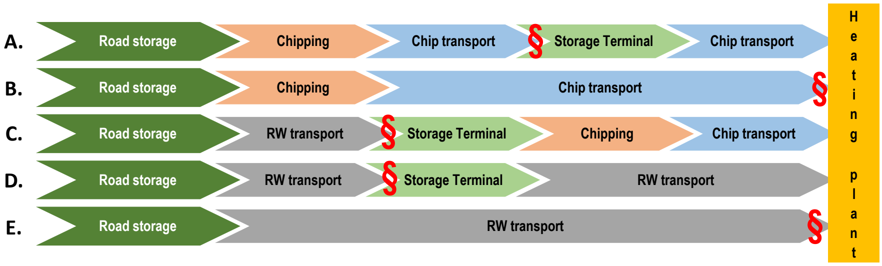

Figure 1. Schematic example of flows (A-E) in forest fuel supply chains in Sweden. The §-sign marks points in the supply chain where measurements are a statutory requirement.

1.2 Forest fuel quality measurements

1.2.1 History of timber quality measurements

Sweden's first timber measurement association was formed in Ådalen by saw timber buyers in 1892, and was later followed by similar associations in the northern, densely forested, parts of the country. In these early associations, sellers had no influence over timber measurement procedures, which were totally controlled by timber buying companies, mainly sawmills (Pettersson, 2011). Growing criticism from the forest owners led to demands for legislation to ensure fair and uniform timber measurement procedures, and the first Timber Measurement Act in Sweden came into effect in 1935.

This law was intended to ensure timber measurement impartiality by establishing timber measurement councils, with equal representation of sellers and buyers, which were responsible for executing payment-determining measurements on coniferous saw timber for industrial use or export (Bäcke et al., 2010). All active timber measurement associations were immediately restructured into impartial associations according to the proposed timber measurement councils. The boards of these new associations comprised equal representation of sellers and buyers, under the guidance of an independent chairman, and this is still applying today.

Today, three regional Timber Measurement Associations (VMF) conduct quality controlled measurements for approximately 97% of all coniferous saw logs and pulpwood traded in Sweden, but only for a small proportion of the traded forest fuels. The Swedish Forestry IT company (SDC) administers the registration of timber contracts, price lists, transports, etc. SDC carries out audits for the timber trade, e.g. volume and price calculations, based on the VMF measurements, and provides measurement results to sellers and buyers.

The timber measurement organisations are economic associations, jointly owned by the forest industry partners (sellers and buyers), and were set up to ensure impartiality in timber quantity and quality measurement and its accounting. The SDC board of directors is appointed by the sellers and buyers to assume overall responsibility for timber quality measurements (SDC, 2009). SDC's responsibility also includes development of new timber quality measurement methods, technology and instructions, but also accreditation of measuring companies, type approval of measurement equipment, and quality control of measurements. The work is organised in two SDC departments: Timber Measurement Development (VMU), and Timber Measurement Control (VMK).

Historically, forest fuel measurements have not been covered by the Timber Measurement Act. Of the 6-10 million cubic metres of forest fuel traded annually, more than 90% was measured by the seller or the buyer (Björklund, 2014; Bäcke et al., 2010). As the demand for forest fuel increased in the early 2000s, the method of business started to change. In the saw timber and pulpwood markets, there are already established trade measures for saw logs (m^3 top measured under bark) and pulpwood (m^3 solid under bark). For forest fuels, many miscellaneous measurement units apply; for example, logging residue chips can be paid in m^3 loose volume of chips, dry tonnes, raw tonnes, energy content per tonne (MWh/tonne), sometimes per harvested hectare or harvested volume of saw timber and pulpwood, or simply an overall price as in purchase of standing timber (Bäcke *et al.*, 2010). Often, fuel quantity and quality was measured by the recipient at the heating plant or terminal, and measurement methods varied greatly; it was up to the agreement parties to decide how measurements should be performed.

1.2.2 Timber Measurement Act of 2015

The new Timber Measurement Act of 2015 was prompted by changes in the timber market, with a strong growth in wood fuel trade and development of alternative trading forms. The Act replaces the Timber Measurement Act of 1966, which was very succinct and included only quality measurement for coniferous saw logs and pulpwood. In 2008, the Forest Agency, which is appointed by the Swedish Government to be the supervisory authority for the Timber Measurement Act, started to revise timber measurement regulation to better correspond with the market and changes in EU legislation (Bäcke et al., 2011; Bäcke et al., 2010). In this process, sellers and buyers were active in consultation and preparation of new draft regulations. The sellers and buyers saw the need for special timber measurement legislation, since legislation promotes credibility, consistent and accurate measurements, quality assurance, and orderly trading in the market, and prevents supply chain disruptions.

One of the more important changes in the 2015 Act is the definition of timber, which now includes all parts of the tree – stem, stump and branches of felled trees – regardless of treatment prior to industrial processing (Anon., 2014b). This means that all woody biomass supplied from the forest for commercial use must be measured, unlike the previous legislation, which only applied to coniferous saw logs and pulpwood. The measurement requirements are limited to the first level of trade, i.e. when a forest owner sells timber and the timber reaches the market. It does not regulate who is to conduct the measurements in any other way than that they must be registered with the Forest Agency as a measurement company. This opened for any parties in a timber transaction to conduct quality controlled measurements.

Consequently, the new Act is more stringent in terms of provisions regarding measurement procedures. The most significant requirements are:

1) The measurement company must submit notifications to sellers and buyers about delivery ID, quantity, assortment, date, location, conducting ID, measurement methods, sellers, buyers, pricelist, etc.

2) The measurement company must have a systematic and effective quality control protocol for measurement equipment and procedures.
3) Only measurement procedures (including any type of conversion figures or functions) and devices that through
    a) documented research results,
    b) documented tests in a practical scale, or
    c) documented experience,

    have been shown to provide satisfactory results with insignificant systematic errors may be used.
4) Only insignificant systematic errors may occur in the completed timber measurement results.
5) Measurements may not deviate from the accuracy requirements set for measurement of weight, volume, energy value, or number of pieces.

Weight is measured either in raw tonnes, by weighing the truck before and after unloading, or in dry tonnes, where moisture content must also be determined. Volumetric measurements are usually taken with the fuel still loaded on the truck. For roundwood, solid volume is determined by stack measurement and, for chips, loose volume is measured. For chips, energy value is usually determined, which requires information on raw tonnes, moisture and ash content. The measurement of number of pieces is not used for forest fuel assortments (Björklund, 2014). To meet the legal requirements, it is not enough to measure the quantity of biomass accurately and with acceptable precision – fuel quality parameters must also be determined accurately and with acceptable precision. As these parameters are often determined on samples taken from the delivered biomass, correct and unbiased sampling procedures are vital.

The new law meant that many more recipient sites were subject to legal requirements for measurement procedures (Figure 1). A survey by SDC/VMU revealed that about 260 storage terminals for forest fuel, and 100-200 heating plants were affected (Björklund, 2014; Björklund & Eriksson, 2013a). At terminals, measurement was usually performed by the truck drivers, measuring loose volume of chips in cubic metres or solid volume for logs. At the heating plants, truck drivers usually provided samples of the delivery to the plant's staff for moisture content determination, and at a few locations plant staff carried out all the measurement procedures. These measurement sites had no coordinated or common procedures for measurement; instead, each terminal or heating plant had its own methods and routines, with rarely any systematic control or follow-up on the measurement procedures and results (Björklund & Eriksson, 2013b).

The forest fuel industry is trying to find solutions to meet new legal requirements; measurement issues have been given high priority at SDC/VMU, the VMF and in the R&D programme, Efficient Forest Fuel Supply Systems (Iwarsson-Wide & Björheden, 2015).

1.2.3 Sampling procedure

In a study by SDC/VMU (Björklund & Eriksson, 2013a), moisture content data produced by the following sampling procedures were compared: 1) Truck drivers took chip samples after unloading with their hands, and 2) as a reference, VMU took 20 evenly distributed samples from the same unloaded chips. Based on 26 deliveries (truck loads), moisture content differed from -17 to +27 pp. The truck drivers' sampling method generated an average overestimation of the truckloads moisture content by 3.3 pp, with a standard deviation of ±11.7 pp. This example shows the significance of sampling, and it is reported that 80% of the error in moisture content determination is due to incorrect sampling (Vikinge & Gustavsson, 2016; Strömberg & Svärd, 2012).

In the new SDC-instructions for VMF sampling procedures, samples can be taken either directly from the truck load or from the chip stack after unloading on the ground (SDC, 2017a; SDC, 2017c). The number of samples to be taken depends on the moisture content variation in the material and the number of truckloads in the delivery, but the recommendation is that six samples be taken per truck load.

The size of the respective samples varies from 0.5-2.5 L and depends on the particle size of the chips, but normal sample size for fuel chips is 1- 2 L. The six samples collected are then combined into a general sample, from which one subsample of 1-2 L is taken for moisture content determination using the oven-drying method (SDC, 2017a; ISO, 2015b).

The following procedures must be applied for sampling:

1) Direct from truck load
    a) samples may be taken if the samples are likely to be representative of the entire truck load
    b) samples must be taken from a measuring bridge
    c) samples must be taken from at least 10 cm below the surface of the load
    d) sampling points should be distributed uniformly or randomly across the surface of the measurement unit, but practical feasibility must be considered, i.e. sampling must not compromise safety.

2) After unloading on the ground
    a) stacks or piles are divided into six equal-sized parts. (Figure 2)
    b) samples are collected from the centre of each of the six parts of the stack/piles
    c) if sampling is not done directly after unloading, samples are taken from at least 10 cm below the surface.

Figure 2. Sampling principle from stack or pile after unloading on ground. Sampling points are distributed around the stack or pile. Picture from (SDC, 2017a)

1.2.4 Determination of dry mass and calorific value

The standard method currently used for determining the mass of dry fuel chips on a truck load is as follows: a) Chips are sampled from the truck load before or after unloading to determine moisture content according to the standard method of oven drying the samples at 105˚C until constant weight is reached, usually for 24 hours or more (SDC, 2017a; SDC, 2017c; ISO, 2015b); b) the truck is weighed before and after unloading and the net weight of raw chips is calculated. In theory, the method is unbiased when representative samples are taken from the biomass, but it is not without potential sources of error, including sampling, sample size, oven drying, and precision of the scales used.

The standard method currently used to determine the net calorific value as received for a truck load of fuel chips is to calculate it from the moisture and ash content, the net calorific value of ash-free dry matter, and the enthalpy of vaporisation of water (SDC, 2017a; SDC, 2017c). Moisture content is determined through oven drying but analysis of the other three entities require laboratory equipment, so are generally determined through low-frequency sampling or taken from tabulated data by agreement between the parties (Björklund, 2014). The use of tabulated values increases the measurement uncertainty and denies the possibility to assess measurement accuracy (standard error) for individual truck loads. However, the most common source of calculated net calorific value errors is moisture content uncertainties (Vikinge & Gustavsson, 2016).

1.2.5 Measuring for production management

Information about both the moisture content and the net calorific value in delivered forest biomass fuel is very important for trade and supply management. Despite this, there is virtually no measurement of these parameters for production management.

The Finnish BEST research project (Sikanen et al., 2016) stated that the main idea of an efficient forest fuel supply chain is that all measurements are performed while the material is being handled or transferred in the supply chain, i.e. when chipped, loaded or unloaded. The important measuring points are: 1) in the forest stand, 2) at harvesting, 3) at forwarding, 4) at roadside storage, 5) at chipping/loading, 6) during unloading and loading at terminal, 7) on delivery to terminal or heating plant.

Today, the produced timber and pulp wood volumes of harvesters and forwarders are usually reported to the Swedish forestry IT company (SDC), which compiles data and delivers it to the actors responsible for the harvesting operations. Some forestry and timber buying companies have internal systems using different, self-developed models for moisture predictions. A more advanced method is to predict the moisture content of the stored, uncomminuted material using prediction models based on historical meteorological data (Routa et al., 2015; Erber et al., 2014), but this has not yet been applied in Swedish forest fuel supply management. One reason for the slow progress of measurement-based forest fuel supply chain control is the low value of the product. However, there is also a lack of cost-effective, fast measuring equipment and methods. If an efficient forest fuel supply system is to be developed, it is necessary to develop and validate new methods for measuring forest fuel quality parameters at high precision while the material is being handled or transferred in the supply chain.

1.2.6 Novel measuring methods, primarily for moisture content

In the Swedish R&D programme, Efficient Forest Fuel Supply Systems (Iwarsson-Wide & Björheden, 2015), measurement issues have been a top priority since 2010, primarily with focus on moisture content determination. In a survey by Sjöström (2011), technical principles for moisture content determination, including an overview of equipment available on the market, were presented.

The most common technique among instruments on the market involved electrical resistance and capacitance. Many of these were originally developed for the sawmill industry, for measuring the moisture ratio in planks and boards (Forsén & Tarvainen, 2000) but the measurement penetration depth was too narrow to be used for fuel chips. There were two types of capacitance meters (container and probe) designed for forest fuels, of which the handheld probe type (Farmcomp, 2017) seemed to be most interesting for further research (Volpé, 2013; Fridh, 2012).

Magnetic resonance was a technology that showed good results on a lab rig (Järvinen, 2013), and dual-energy x-ray absorptiometry was also promising (Hultnas & Fernandez-Cano, 2012; Kullenberg et al., 2010), but only for wood chips and not for logging residues. Near-infra red spectroscopy has been used for many years for measurements in the food industry and for agricultural crops, and with good results for some forest fuel assortments (Leblon et al., 2013; Jensen et al., 2006; Lestander & Rhén, 2005; Thygesen & Lundqvist, 2000a). Some of the techniques, like gamma spectroscopy, can provide high accuracy, but the radiation risk and the high safety requirements make them unsuitable for use in forest fuel measurement.

Other techniques possible were ultrasonic, microwave (Senfit, 2017) and radio frequency spectroscopy (Inadco, 2017; Fernandez-Lacruz & Bergström, 2016). Many techniques and instruments were available on the market, but few were designed for application to forest fuel, or even impartially tested for accuracy and precision.

In the period 2013-2015, three specially designed instruments for biomass fuel measurements appeared on the market, and seem to be very interesting. They were marketed as being fast and accurate (Valmet, 2017), fast, accurate and suitable for frozen materials (Prediktor, 2017), and fast, accurate, for frozen material while simultaneously detemining ash, moisture content and calorific value (Mantex, 2017b).

These three instrument technologies – magnetic resonance, near-infrared, and X-ray – together with the handheld capacitance probe instrument were the subject of a more extensive research and development project. This thesis is the result of that R&D project.

2 Objectives

The overall objectives of the studies on which this thesis is based were to study and validate measuring instruments with different technical principles for determining forest fuel quality parameters, and their operational applicability in the forest biomass fuel trade.

The specific aims were as follows:

- Develop a statistically robust validation model for measurement precision and accuracy, where the results are comparable regardless of quality parameter, biomass material, measurement technology principle, instrument model, or individual instrument (Papers I-IV).

- Study and validate the accuracy and precision of:
    i. moisture content when measuring stem wood chips using a handheld electric capacitance instrument in an operational environment (Paper I)
    ii. moisture content when measuring non-frozen forest fuel materials using a nuclear magnetic resonance instrument (Paper II)
    iii. moisture content when measuring both frozen and non-frozen forest fuel materials with a near-infra red spectroscopy instrument (Paper III)
    iv. net calorific value, ash and moisture content when measuring both frozen and non-frozen forest fuel materials with a combined dual-energy x-ray absorptiometry and fluorescence spectroscopy instrument (Paper IV).

- Analyse the possibility to determine particle size distribution, median particle size, and fines of sampled chips by multivariate analyses and modelling using data from the X-ray instrument studied in Paper IV (Paper V).

- Analyse and provide suggestions for methodology and application of the studied instruments with the aim of minimising sampling errors.

3 Methodology

The studies and validation of the measuring instruments are described in detail in Papers I-V. A summary of measuring techniques, biomass fuel materials and quality parameters, number of samples, reference measurements, and instrument measurements used in that research is presented in Table 1.

Table 1. *Overview of measuring techniques, biomass fuel materials, quality parameters, and number of unique samples, reference and instrument measurement values in the studies described in Paper I-V.*

Studies included in this thesis			*Paper*				
			I	II	III	IV	V
Measuring techniques							
Electric capacitance (CAP)			X				
Magnetic resonance (MR), two instruments (MR-C & MR-S)				X			
Near-infrared spectroscopy (NIR)					X		
Combined dual-energy x-ray absorptiometry & fluorescence spectroscopy (CXR)						X	X
Biomass fuel material							
Stem wood chips			X	X	X	X	X
Logging residue chips				X	X	X	X
Sawdust					X		
Bark					X		
Biomass fuel quality parameter							
Moisture content (%)			X	X	X	X	
Net calorific value (MWh/ton)						X	
Ash content (%)						X	
Particle size class (P-class)							X
Median particle size (mm)							X
Fines (%)							X
Total number of							
Unique samples	Σ	446	160	66	100	60	60
Reference measurement values	Σ	926	200	86	100	180	360
Instrument measurement values	Σ	211 585	500	425	500	2 160	208 000

3.1 Measuring techniques

3.1.1 Electric capacitance

Moisture content has a major influence on the dielectric properties of wood (Skaar, 1988). Consequently, the moisture content of forest biomass chips can be determined by measuring the electric capacitance (CAP) represented by the dielectric constant, which then can be related to a specific moisture content by constructing a calibration curve. The electric capacitance is difficult to measure using a direct current voltage, so an alternating current (AC) voltage is used in the measuring instruments. If an AC voltage is applied across two conductors separated by an insulating material, e.g. wood, there will be a difference in electric charge between the two sides (Figure 3). Electric capacitance is the relationship between voltage and the difference in charge, and different insulating materials, such as wood chips, bark, and sawdust, provide various electrical capacitance values. The dielectric constant of a material is the ratio of the capacitance of vacuum and the capacitance of the considered insulating material, V_a/V_b (Figure 3).

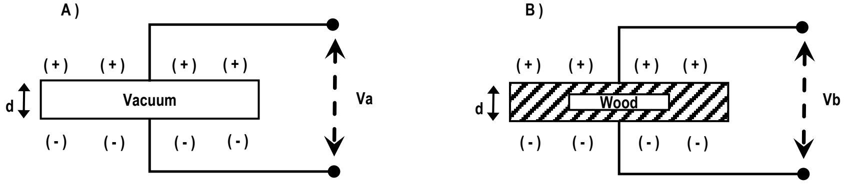

Figure 3. Schematic figure illustrating electric capacitance. Charge accumulation (+ and -) on two conductors at a distance d apart for: A) an AC voltage V_a in vacuum, B) a lower AC voltage V_b with a wood dielectric material. The wood material in B) requires a separate voltage V_b for the charge difference (+ and -), to be equal to the charge difference for vacuum, at distance d.

The dielectric constant generally increases with increasing moisture content, and wood density (Jensen et al., 2006), and reduced frequency. It also increases with increasing temperature except at high moisture levels, where it decreases with higher temperature. Since the dielectric constant is similar for ice and for wood, capacitance moisture meters should not be used on frozen wood chips. The capacitance instrument used in this thesis was a Wile Bio Moisture Meter (Farmcomp, 2017). The instrument was studied in a test lab and in an operational field test. More details about the instrument and the study can be found in Paper I.

3.1.2 Magnetic resonance

Nuclear magnetic resonance (MR) works at the interface between an external magnetic field and the nuclear magnetic moments in sample material. If a sample is placed in an external magnetic field and simultaneously exposed to electromagnetic radiation, it absorbs energy at a certain frequency equalling nuclear precession movement. The nuclei of hydrogen is easy to detect, so MR is best applied for analysis of substances containing a lot of hydrogen, i.e. moisture in biomass fuels (Järvinen, 2013).

An MR moisture measurement device consists of a magnet (coil and housing), a sample container, a scale, a RF radiofrequency (RF) coil and pulse generator, and a receiver (Figure 4A). The MR moisture content determination method is based on the fact that the hydrogen atom spin makes it a magnetic dipole, a small magnet. Exposing a sample, which is already in a static magnetic field, to a short and powerful dynamic electromagnetic field (RF-pulse) perpendicular to the first field excites the magnetic dipoles. After excitation, precession of the magnetic dipoles induces an electric field that drives a current to the coil wound around the sample (Figure 4 B-F).

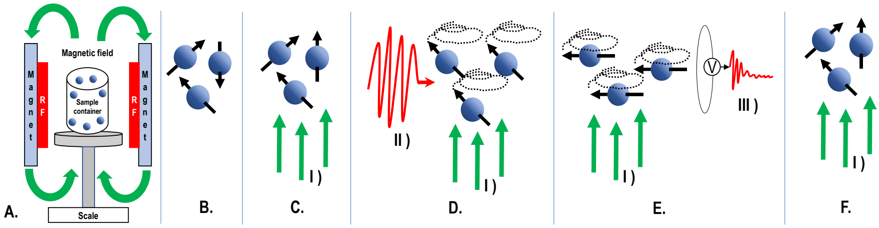

Figure 4. A. Schematic instrument configuration with scaler, sample container, magnets creating magnetic field, and the RF-generator/receiver. B. Nuclei spin randomly oriented – no magnetic field. C. Spins aligned by strong magnetic field (I). D. Coherent excitation by the 90° RF-pulse (II). E. Recording of the induced spin RF-eco (III), F. Realignment and loss of coherence. (Figure after Metso MR)

The induced voltage in the coil, often called Free Induction Decay (FID), has been shown to increase linearly with sample moisture content. In addition to the nuclei of hydrogen atoms in water, protons in the wood also induce a voltage, but the time span varies between the two. Hydrogen nuclei in water have a 'relaxation time', i.e. the time from excitation to realignment (Figure 4 D & E), longer than 300 µs, while wood has a 'relaxation time' of approximately 15 µs. Measuring the voltage at a carefully chosen time interval after the sample has been exposed to pulse will screen out the wood proton effect.

The MR instrument analyses the weight of the water in the sample container, weighs the total sample mass, and from that, calculates the moisture content in percentage points. One drawback with the MR technique is that it cannot be used to measure moisture content in frozen material (Sjöström, 2011). The MR study in this thesis was performed with two magnetic resonance instruments, MR-S and MR-C, of the same model Valmet (former Metso) MR Moisture Analyzer (Valmet, 2017). MR-S was studied in a test lab and MR-C in an operational environment. More details about the instrument and the study can be found in Paper II.

3.1.3 Near-infrared spectroscopy

Near-infrared (NIR) energy is the electromagnetic energy of molecular vibration, and NIR-spectroscopy is a technique based on the ability of chemical bonds to absorb radiation at certain wavelengths specific to a given material. It measures either the transmittance or the reflectance of NIR radiation by a solid, liquid, or gaseous sample material. The electromagnetic spectrum for the invisible NIR light has a wavelength from 700 nm to 2500 nm, while the wavelength for visible light ranges between 400 nm and 700 nm. Water displays specific bands at approximately 1440 nm and 1940 nm (Siesler et al., 2008). NIR spectra are normally collected in reflectance mode, but often shown as NIR absorbance, which is defined as $\log_{10}$ [1/reflectance] (Leblon et al., 2013) The NIR-spectra is affected by a variety of factors, such as moisture content, temperature, and the biomass materials density, so it has to be calibrated for these factors.

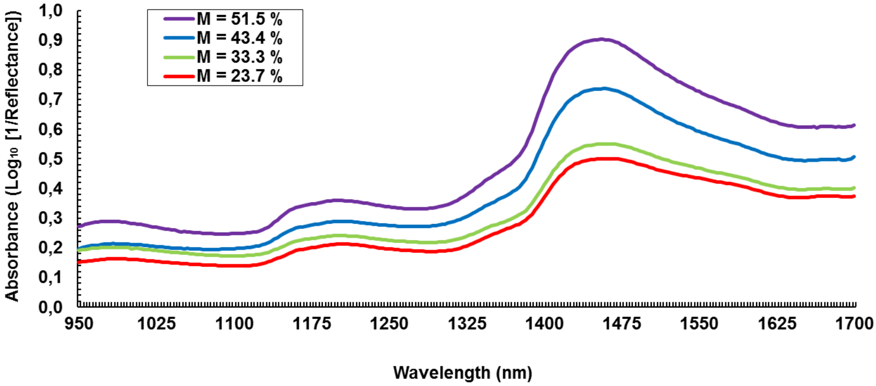

Figure 5. Four NIR-spectra from measurements of logging residues samples, ranging from 23.7% to 51.5% reference moisture content.

Moisture content is determined by a calibration technology called Real Time Calibration (RTC), based on calculating the relationship between the reference values of the physical samples and the NIR measurements. For every new measurement, the NIR spectrum is compared with the NIR spectra of the samples contained in the calibration matrix. A new, local model for moisture is generated in real time. The model is based only on the NIR spectra in the calibration matrix. The moisture peak position is located at approximately 1440 nm for non-frozen and frozen biomass chip material (Figure 5). The main advantage of the NIR spectrometer technology is the potential to differentiate between the peak absorption wavelength (spectra) of frozen versus unfrozen material to measure moisture content by utilising the best fitting spectra. The NIR-instrument used in this study was a Prediktor Spektron Biomass (Prediktor, 2017) and was studied in a test lab. More details about the instrument and the study can be found in Paper III.

3.1.4 Combine dual-energy x-ray absorptiometry and fluorescence spectroscopy

Dual-energy X-ray absorptiometry (DXA) and X-ray fluorescence spectroscopy (XRF) can be combined (CXR), and processing by multivariate image analysis, image feature generation extraction, and data fusion enables prediction of forestry fuel quality parameters. The DXA quantifies the fraction of emitted X-ray photons that passes through a sample when irradiated with photons with two different mean energies.

Attenuation of X-ray photons in a material depends on three different factors: i) X-ray photon energy, ii) sample thickness, and iii) sample material. All basic elements have a specific attenuation that is a function of the incident photon energy. How a sample attenuates X-ray photons at one energy level (monoenergetic) can be calculated using a mathematical model (Beer-Lambert relationship) as:

$$\mathrm{I} = I_0 e^{-udC} \qquad (1.)$$

where I_0 is the number of photons before, and I the number after, the beam has passed through the sample material, d is the material thickness, u is the energy and material dependent attenuation constant, and C is the molar concentration of the sample substance. The difference in a material’s attenuation characteristics allows measurements of sample properties at the atomic level (Heismann et al., 2003).

The photon transmission rate ($\mathrm{I/I_0}$) from Equation 1 results in two equation systems, one for each energy, which makes it possible to solve the concentration of two different chemical components, carbon and oxygen, given that everything else in the sample is relatively constant. The carbon and oxygen levels are then used to determine the moisture content based on the assumption that the oxygen content in wood is relatively constant. Differences in oxygen level are therefore derived from the changes of water content. Other atoms, primarily ash-forming elements, disrupt DXA measurements and significantly impair the accuracy of moisture content and net calorific value measurements. To overcome this problem, XRF is utilised by adding information on ash-forming element content in the sample (Torgrip & Fernández–Cano, 2017). XRF measures the abundance and energy of fluorescent photons emitted by atoms irradiated with X–rays as a secondary effect. Energy from emitted photons has a characteristic pattern that is unique for each chemical element, and this enables identification of all atoms with an atomic number greater than 12 in the periodic table (Figure 6).

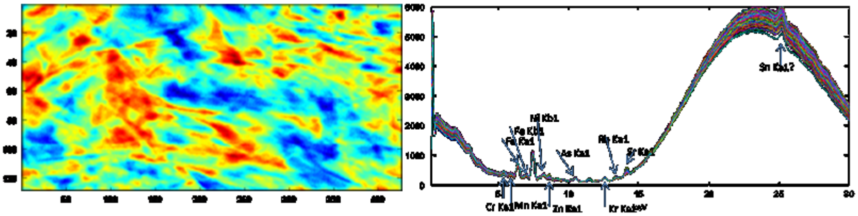

Figure 6. Left: DXA image showing the photon attenuation (blue is high and red is low) of a forest residue sample. Right: XRF data from 40 wood chip samples with a range of energy peaks at positions corresponding to each of the elements in the sample. The peak integrals (height) is proportional to the element concentration. (Pictures by Mantex AB)

The obtained emission of XRF photons creates a spectrum of energy peaks at positions that correspond to each of the elements in the sample, and the peak integrals (height) are proportional to the concentration. The number of atoms that interfere with the DXA sensor, i.e. ash content and ash composition, is calculated from the XRF data. XRF analyses the surface of the sample, not the bulk of it, so the use of XRF data for calibration assumes that the surface measurements are representative of the bulk of the sample. The main advantage of the DXA-XRF combined configuration is that it can measure both frozen and non-frozen materials, and its calibration allows determination of moisture and ash content and net calorific vale at the same time. The X-ray instrument used in this thesis was a Mantex Biofuel Analyzer prototype (Mantex, 2017b) and was studied in a test lab. More details about the instrument and the studies can be found in Paper IV and V.

3.1.5 Reference analyses

Reference analyses were conducted on all samples and for the entire sample. The size of the samples ranged from 0.8 L to 13 L.

Reference moisture content was determined by the oven-drying method according to the Solid Biofuel standard, EN-14774-2 (CEN, 2009) in Paper I-III and ISO 18134-2 (ISO, 2015b) in Paper IV. The differences between the two standards are small and it is basically the same method. The reference moisture content for each sample was calculated as:

$$\text{Moisture content} = 100 \, * \, \frac{(mass_{wet\ sample} - mass_{oven\ dry})}{mass_{wet\ sample}} \qquad (2.)$$

Ash content and gross calorific value (in Paper IV) were determined by a commercial laboratory according to ISO 18122 (ISO, 2015a) and EN 14918 (CEN, 2010). Reference net calorific value as received was calculated according to functions for net calorific value at constant pressure as stated in EN 14918, section 12 (CEN, 2010). In Paper V, particle size distribution and median particle size (d50) were determined by sieving according to ISO 17827-1 (ISO, 2016) and the particle size class and the fine fraction were specified according to ISO 17225-1, Table 5 (ISO, 2014).

3.2 General validation model

3.2.1 Prerequisites and requirements for the validation model

A validation model was needed to evaluate the instruments with different measuring techniques. The model had to be statistically robust, results had to be comparable, regardless of the studied quality parameter, biomass material, measuring technique, instrument model or individual instrument. The validation model had to provide answers about the studied instruments' measurement accuracy and precision in comparison with the reference method. Sampling error had to be eliminated by determining reference values for each sample and then comparing them with the instruments value for the same sample. When determining moisture content using the average value of several samples, there will be a sampling error that adds to the actual measurement error of instrument. This sampling error will be equally big for a validated instrument as for the reference method, if the sampling is performed in the same way for both methods. Primarily, the validation model was intended for measurements of moisture content (Papers I-IV), and secondarily for measurements of ash content and net calorific value (Paper IV).

The definition of accuracy and precision are shown schematically in Figure 7. The accuracy refers to the deviation between instrument value and the reference method value (Equation 3), where a sample of small deviations provide high accuracy. The precision refers to the instrument's repeatability, i.e. the ability to provide the same value for repeated measurements on the same sample, where large sample variation provides low precision. The most desirable outcome is a measuring instrument that combines high accuracy with high precision. An instrument with low accuracy but high precision indicates that it is stable but not properly calibrated. This bias may be managed by calibrating the instrument. The worst and most undesirable outcome is when both accuracy and precision are low. The random error cannot be calibrated and the uncertainty of the measurement remains high.

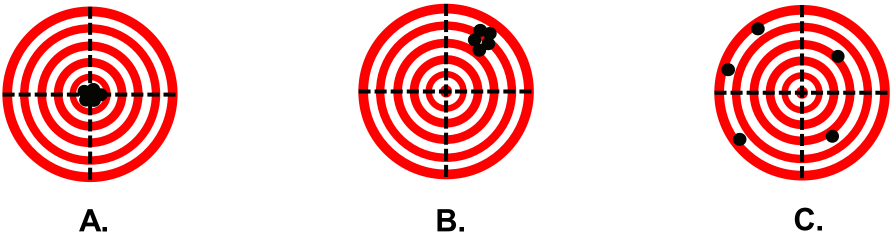

Figure 7. Definition of accuracy and precision: A) High accuracy and high precision is the preferred result; B) Low accuracy but high precision, a systematic error (bias) that can be managed; C) Low accuracy and low precision, an unacceptable result.

The validation model also had to show whether the instrument's accuracy and precision were sensitive in terms of:

1. Type of forest biomass fuel material. The aim was to primarily study the assortments covered by the new Timber Measurement Act: chips from 1) stem wood and 2) logging residues, and secondary residues from the forest industry, such as sawdust, and bark.

2. Moisture content level. The aim was to study whether the instrument's accuracy and precision were sensitive to the moisture content level of the material being measured. Measurements were conducted on samples covering the moisture content range 20-60%.

3. Moisture condition. The aim was to study how measurements were affected by the material being frozen or non-frozen. Measurements on frozen material were conducted with NIR and CXR instruments (Papers III and IV).

Analysis of variance (ANOVA) tests the hypothesis that the means of two or more populations are equal, most commonly under the assumption of homogeneous population variance. ANOVAs assess the effect of one or more factors by comparing the response variable means at the different factor levels.

Since repeated measurements on the samples are to be done, the observations within sample are expected to be more correlated than observations between samples. This means that the model becomes longitudinal to its character, so a mixed-model design ANOVA was the most appropriate.

The mixed model or linear mixed model is a natural extension of the general linear model by allowing the addition of random effects where the levels of the factor represent a random subset of a larger group of all possible levels. The flexibility in modelling the random error and random effect variance components is one of the most important advantages of the mixed model over the general linear model (Engstrand & Olsson, 2003). Notable is that the general linear model's assumption of homogeneous variance is not necessary for the mixed model. Another strength is that it allows the modelling of both heterogeneous variances and correlation among observations by specifying the covariance structures for the unknown random effects and the unobserved random errors. To improve the accuracy of the fixed effect estimates, covariates (continuous) and/or nested effects may be included. It is then important to specify an appropriate covariance structure for the model, since the hypothesis tests treatment mean estimates, and confidence intervals are all affected by the model's covariance structure. The variance matrix estimates are obtained using restricted maximum likelihood (REML). The fixed effects in the mixed model are tested using F-tests with the following three assumptions: 1) the response variable is continuous, 2) the individuals are independent, and 3) the random error follows the normal probability distribution with mean equal to zero.

3.2.2 Sample preparation and measurement procedure

The forest biomass fuels used in the studies of this thesis belonged mainly to two different assortments: chipped stem wood (SW) and chipped logging residues (LR). The SW chips came from non-barked low-quality logs that did not meet forest industry quality requirements. The material was dominated by spruce (*Picea abies*) logs, mixed with small amounts of pine (*Pinus sylvestris*) and hardwood logs, mainly birch (*Betula spp.*). LR consisted of chipped branches and tops, mainly from spruce with small amounts of pine and a mix of common hardwoods such as birch and aspen. This division of assortments is rather approximate – there are large variations with respect to moisture content, ash content, particle size and density within each assortment, especially for LR. However, they were chosen since SW chips and LR chips are the dominating assortments used in forestry fuel trading today, so are significantly affected by the requirements of the new Timber Measurement Act.

The material was collected randomly, at the supplier's storage terminals and at the fuel reception, control, or storage site at the heating plants. The aim was to obtain samples in the moisture range of 20-60%. A handheld capacitance meter was used to obtain a rough estimate of the initial moisture content in the field. Collected materials were put into sealed 30-70 L plastic boxes and transported to the lab where they were stored in a refrigerator until further sample preparation. NIR measurements were also taken on the two forest industrial residue assortments of sawdust and bark (Paper III). MR measurements were also performed on pulpwood chips (bark-free), stem wood fuel chips, bark, and mixed fuel, comprising an equal mix of all three discussed above (bark, biomass and pulp chips) (Paper II).

The objective was to prepare a minimum of five measurement samples within each moisture content class (Table 2) for each assortment. When the collected materials did not cover the entire moisture content range, samples had to be further prepared in the lab. Materials in M_CL 2 and M_CL 3 were difficult to find, so moist material was dried in the oven between 30 and 140 minutes to ensure sufficient measurement samples in all moisture classes, covering the entire range 20 to 60%. After lab preparation, all the samples were stored either in a refrigerator (+4°) or in a freezer (-23°C) until the measurement procedure.

Table 2. *Definition of moisture content classes (M_CL).*

Moisture content class (M_CL)	Moisture content (M) %
M_CL 2	20.0 - 29.9
M_CL 3	30.0 - 39.9
M_CL 4	40.0 – 49.9
M_CL 5	≥ 50

In each study, measurements were performed starting with one assortment, conducting both the test and the reference measurement before moving on to the next assortment. The general procedure was as follows. A random sample was selected and exposed to the instrument. Five repeated measurements were taken and the reading from the instrument was recorded. The next sample was then measured and the procedure repeated until all samples were measured. The reference values of the samples were then determined. For each assortment, there were at least five samples in each of the four moisture content classes. Five repeated measurements per sample meant that a minimum of 100 measurements per assortment were taken. This general procedure had to be adjusted and fine-tuned depending on the technical conditions for each instrument. For further details of the exact procedure used with respective instrument see Papers I-IV.

For the CAP instrument, measurement was carried out on samples in four measuring cycles, and the samples were dried gradually between each cycle. This method was chosen to minimise the impact of density, by performing measurements on the same material but at different moisture content levels (Paper I). A field study was also carried out. Based on the lab test, an attempt was made to create a calibration function with a polynomial regression model that was then used in the field test.

For the NIR and CXR instruments, measurements were also performed on frozen materials. The CXR instrument used standard plastic containers with a tight lid, which were supplied together with the instrument by the manufacturer. These were used to store and handle each chip sample. The same sample could therefore be measured in both frozen and non-frozen conditions without further preparation (Paper IV). For the NIR instrument, samples were divided into two equal parts, one measured unfrozen and the other non-frozen. Making two separate samples was needed because the measurements had to be performed in a 5 L open tray and the material had to be mixed between each repeated measurement. If this procedure had been conducted for the same sample, first frozen and then non-frozen, the material would have started to dry out and the reference moisture content would have been different for the two measurements (Paper III).

3.2.3 Statistical analyses

The statistical analyses were carried out for each instrument separately, using SAS Enterprise Guide 6.1 (SAS Institute Inc., NC, USA) and Statistica 13 (Dell Inc., TX, USA). The analytical model described here is a general model. In the various studies (Paper I-IV), some small adjustments were made based on the measurement procedure established for each individual measurement method.

To analyse the measurement accuracy, the difference between instrument measurements (INST) and reference methods (REF), in moisture content (DIFF_M), ash content (DIFF_A) and net calorific value (DIFF_q) was calculated as:

$$\mathrm{DIFF\_X_{ij}} = \mathrm{INST\_X_{ij}} - \mathrm{REF\_X_{i}} \qquad (3.)$$

where X is equal to moisture content (M), ash content (A), or net calorific value (q) in their respective analysis, i is sample identity, and j the repetition of measurements using the instrument on each sample. In the analysis, all samples were subdivided into moisture content classes based on their reference moisture content (Table 2). Using this categorised value for moisture content instead of continuously distributed values removes covariance structures that may affect the reliability of model estimates.

The following mixed linear model, containing both fixed and random factors, was used for each differential analysis:

$$DIFF\_X = \alpha + \beta + \gamma + \alpha * \beta + \alpha * \gamma + \beta * \gamma + \alpha * \beta * \gamma + a + \varepsilon \qquad (4.)$$

where DIFF_X is equal to DIFF_M, DIFF_A, or DIFF_q in their respective analyses. Fixed factors were forest biomass fuel materials (α), moisture content class (β), and moisture condition frozen/non-frozen (γ), whereas sample identity was used as a random factor (a) and ε is a random error term. Fixed factor significance levels were tested with a Type III test, where effects were considered significant if $p < 0.05$. Tests concerning linear combinations of least square means in general, including Type III tests concerning differences of least squares means, are considered independent of the parameterisation of the design. This makes Type III sums of squares useful for testing hypotheses for unbalanced ANOVA design with no missing cells, as well as for any design for which Type I or Type II sums of squares are appropriate (Engstrand & Olsson, 2003). Type III test provides the sum of squares that would be obtained for each variable if they were entered last into the model, so the effect of each variable is evaluated after all other factors have been accounted for. Therefore, the result for each term is equivalent to that obtained with Type I analysis when the term enters the model as the last one in the sequence.

Another strength is that the test is independent of sample size. The effect estimates are not a function of the frequency of observations in any group (i.e. for unbalanced data, where we have unequal numbers of observations in each group). When there are no missing cells in the design, these subpopulation means are least squares means, which are the best linear-unbiased estimates of the marginal means for the design. Least square means with a 95% confidence interval were used to estimate the mean differences for significant effects.

To analyse the precision (i.e. the repeatability for the instrument), the variance for the repeated measurements within samples was used to calculate the variance between samples from which a 95% confidence interval was calculated.

3.2.4 Modelling X-ray data for predicting particle size variables

In Paper V, the possibility to predict particle size variables of sampled chips was analysed by applied multivariate data analyses and modelling (Everitt & Dunn, 2001) of CXR spectral data from 720 measurements in Paper IV. Since the DXA detector has 512 x 128 pixels and the XRF detector has 1 x 2048 pixels, and each sample was radiated twice (high and low energy), the total number of variables for each measurement was 133 240 with a large proportion strongly correlated. This data set was too large to handle within the objectives of this study. The R&D staff at the manufacturer were asked to process data using the same methods as in their commercial development work. Image feature generation extraction enabled the CXR raw data to be extracted from an n-dimensional data to a vector data set of 290 variables for 720 measurements of biomass chip samples (Table 3). The aim was to try fit a general regression model to predict each of the response variables: particle size class (P), median particle size (d50) and fines (F).

The multivariate methods used were principal component analysis (PCA), cluster analysis (CLA), multidimensional scaling (MDS), partial least squares regression (PLS) and a general regression model (GRM) (Everitt & Dunn, 2001). All analyses were performed with Statistica 13 (Dell Inc., TX, USA). PCA was used to find a few linear combinations of the original variables that can be used to summarise a set of data with as little information loss as possible. The method transforms a set of correlated data to a new set of uncorrelated variables used to provide informative plots of the data. CLA was used as an exploratory analysis to see if or how specific variables were sorted into small groups or cluster. MDS was used to determine both the dimensionality of the model, i.e. how many (n) dimensions provided a satisfactory fit, and the positions of the points in the resulting n-dimensional space. PLS was used as an exploratory analysis tool to select suitable predictor variables as an input to the General Linear Regression model. More details on the analysis and modelling of the CRX data can be found in Paper V.

Table 3. *Variation in particle size classes (P), median particle size (d50) and fines (F) for the collected samples of different biomass chips based on the reference measurements on each sample.*

	Stem wood chips	Logging residue chips
Number of samples	30	30
Sample size, L	3	3
Particle size classes (P)	P31, P45, P63	P16, P31, P45
Median particle size (d50) mm	14.4 – 23.1	7.1 – 14.4
Fines <3.15 mm (F) %	1.5 – 4.2	5.0 – 23.1

4 Results

4.1 Moisture content

4.1.1 Precision

The measurement precision of the instruments, i.e. the ability to provide the same value for repeated measurements on the same sample, were quite similar for all technologies, as shown in Figure 8, except for CAP. All the other instruments had 95% of their measurements within less than ± 2.5 pp.

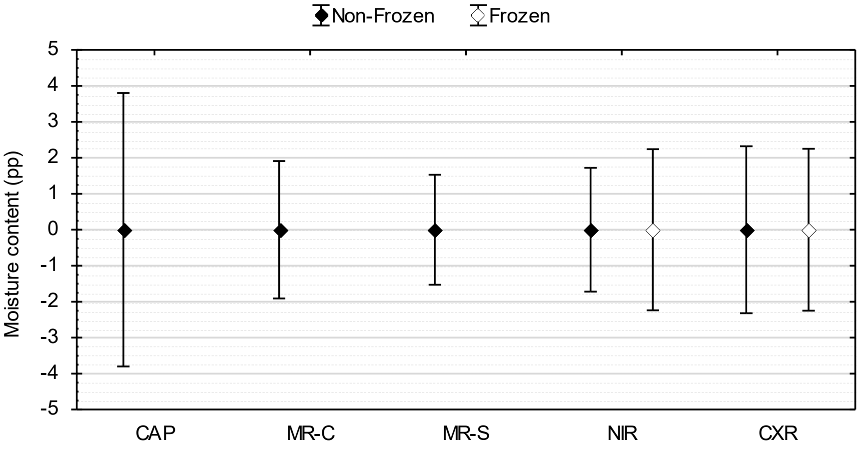

Figure 8. Precision in moisture content measurements presented as a 95% confidence interval of the mean. The markers show the mean and bars the min and max in a 95% confidence interval.

4.1.2 Sensitivity

The effects of the fixed factors and their interactions were tested with an F-test to clarify the effects of factors on the measurement accuracy of the instruments. The MR instruments were affected differently by the interaction between SORT and M_CL. The interaction effect was significant for MR-S but not for MR-C (Table 4). The MR-S had a significant difference between the SORT in M_CL 5, but not in the other moisture classes. This could indicate that the MR technology is sensitive to the type of material in certain moisture content levels, contradicting the theory of MR technology. This had to be further investigated. Both NIR and CXR had a significant impact in the three-factor interaction (SORT x M_CL x M_CON), which needed to be addressed first. This indicates that the instruments are sensitive to the material at different moisture content levels and moisture conditions, and had to be further analysed for each material.

Table 4. *F values (F) and significance probability value (p) for the tested effects of the mixed model fixed factors on the difference in moisture content between the instrument measurement and the reference method. Tested effect were considered significant if p<0.05*

	CAP		MR-C		MR-S		NIR		CXR	
Mixed model factor effects	F	p	F	p	F	p	F	p	F	p
Material (SORT)	x	x	0.75	0.5249	0.95	0.3311	2.30	0.0766	0.11	0.7370
Moisture content class (M_CL)	4.01	0.0087	5.45	0.0005	3.78	0.0054	1.26	0.2882	0.98	0.4015
Moisture condition (M_CON)	x	x	x	x	x	x	1.04	0.3087	118.21	<.0001
SORT x M_CL	x	x	0.25	0.9857	9.51	<.0001	3.23	0.0072	2.10	0.1231
SORT x M_CON	x	x	x	x	x	x	3.28	0.0211	18.33	<.0001
M_CL x M_CON	x	x	x	x	x	x	3.76	0.0241	6.49	0.0003
SORT x M_CL x M_CON	x	x	x	x	x	x	5.01	0.0071	5.94	0.0028

4.1.3 Accuracy

The moisture content measurement accuracy showed large variations, both between instruments, but also between moisture content classes, moisture conditions and the forest fuel materials. An overview of the differences between the instruments at different moisture conditions for two combinations of materials and moisture content classes is given in Figures 9 and 10. The MR-instrument showed the highest measuring accuracy both for stem wood chips and logging residue chips. There was a significant difference between the two instruments, where MR-C on average underestimated the moisture content significantly by 1.84 pp while MR-S underestimated by 0.23 pp. The MR-C was very accurate for moisture content level greater than 50% (M_CL 5) but the underestimation increased with decreasing moisture content level. The MR-S was very accurate and stable, and it was only in M_CL 5 for logging residue chips that a significant deviation greater than 1.5 pp was noted (Paper II).

The CAP instrument, which was tested only for stem wood chips, showed the lowest measurement accuracy. On average, CAP underestimated the moisture content by 6 pp. In M_CL 2, 3 and 4 the underestimation varied between 4-6 pp and for M_CL 5 it drastically dropped to an underestimation of 12 pp. The results confirmed the assumption that calibration was necessary. Since the nominal CAP moisture values were of a similar magnitude in M_CL 4 and 5, it was decided to restrict the calibration of M_CL 2 to 4. A polynomial regression with backward selection and stopped at a minimum of the Mallows C(p) criterion resulted in a calibration function where M_CL 2 to M_CL4 on average was overestimated by 1.8 pp in the field test (Paper I).

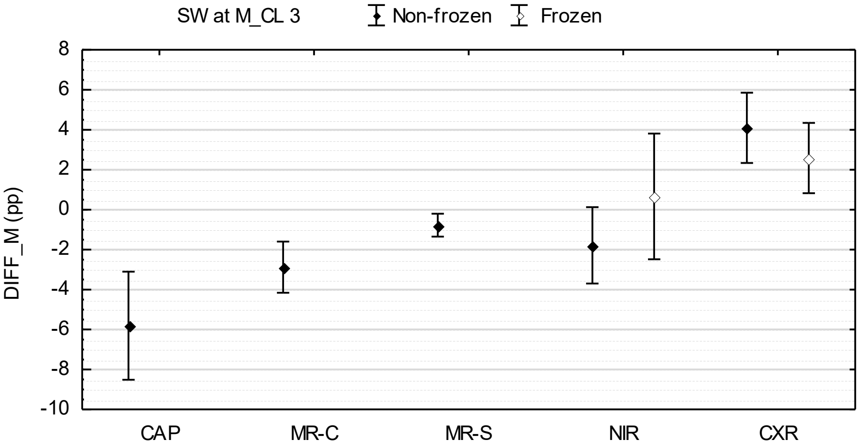

Figure 9. Difference in moisture content (DIFF_M) between instruments and the reference method for stem wood (SW) chips in moisture content class 3 (30.0-39.9%) in both non-frozen and frozen moisture condition. The markers show the least squares mean and error bars a 95% confidence interval.

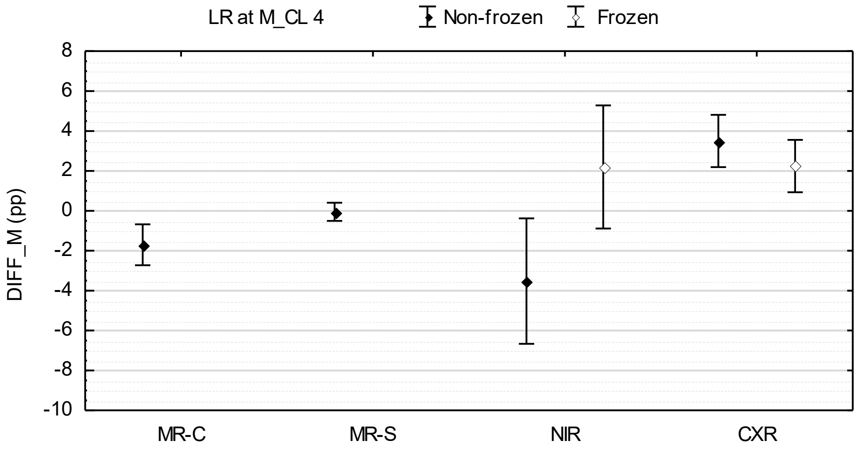

Figure 10. Difference in moisture content (DIFF_M) between instruments and the reference method for logging residue (LR) chips in moisture content class 4 (40.0-49.9%) in both non-frozen and frozen moisture condition. The markers show the least squares mean and error bars a 95% confidence interval.

NIR measurements were performed on both frozen and non-frozen material. On average the accuracy was very good, with less than a 1 pp significant deviation for frozen material and underestimation of 0.34 pp, and for non-frozen material an overestimation by 0.68 pp. Measurements were accurate and consistent for both stem wood chips and logging residue chips. Only for M_CL 4 was a significant underestimate of non-frozen logging residue chips and frozen stem wood chips found (Paper III).

The CXR instrument significantly overestimated moisture content by 1.9 pp for stem wood chips and by 2.4 pp for logging residue chips. For stem wood chips, a significant difference between frozen and unfrozen material was found, where unfrozen chips in M_CL 3 were overestimated by 0.6 pp and then decreased with increasing moisture content class to an underestimation of 1.8 pp in M_CL 5. For logging residue chips, measurements were more consistent with good accuracy in M_CL 3 but with systematic overestimation in the other moisture content classes (Paper IV).

4.2 Net calorific value and ash content

The CXR instrument was calibrated for simultaneous measurement of the ash content and net calorific value, in addition to the moisture content. The CXR performed consistently for all frozen and non-frozen material by systematically underestimating the net calorific value (Figure 11a). On average, the net calorific value was underestimated by 0.53 MWh/ton (21%) for logging residue chips and by 0.33 MWh/ton (12%) for stem wood chips. The precision was within a 95% confidence interval, ± 0.13 MWh/ton for logging residues and ± 0.19 MWh/ton for stem wood chips. The accuracy for the ash content determination was high, with no significant deviations (Figure 11b). The 95% confidence interval for repeatability of measurements on the same sample i.e. the precision, was for both stem wood and logging residue chips ± 0.08 pp of the average ash content (Paper IV).

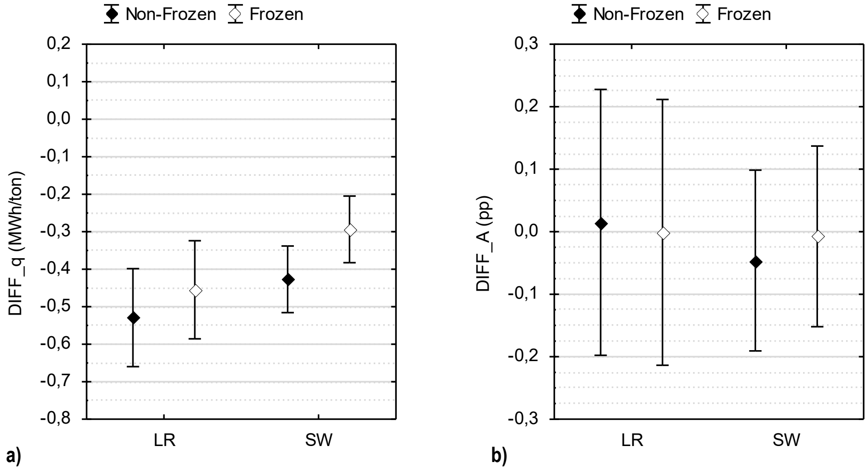

Figure 11. Difference between CXR and reference method in: a) net calorific value (DIFF_q) and b) ash content (DIFF_A) for logging residue chips (LR) and stem wood chips (SW) in both non-frozen and frozen moisture condition. The markers show the least squares mean and error bars a 95% confidence interval.

4.3 Particle size parameters

The analysis revealed that the CXR-data were longitudinally structured within sample boxes, and the mean values of CXR-data were thereafter separately calculated for the respective cases of frozen and non-frozen samples, resulting in a new data set of 290 variables times 120 measurements that was used for further analyses. When this data-set was tested for main effects, the dichotomic factor SORT effectively split the sample into two significantly different classes of stem wood and logging residue chips. The results from the PCA showed that according to the scree criteria, four factors would provide sufficient approximation of total CXR-data variance for each chip material. The analyses of multidimensional scaling resulted in both stem wood data and logging residue data spanning a five-dimensional space. This information was then used in the

PLS, to select the most important variables for each chip material. The top 12 variables were then entered into a general regression model, one for each response variable and material. The results of the GRM are shown in Tables 5-6. The results showed that it was possible to predict the particle size variables by regression function with between 4-8 variables from CXR-data (Paper V).

Table 5. *Basic statistical properties from general regression model for logging residue chips (LR) predicting particle size variables.*

Quality type	Adjusted R^2	df Model	MS Model	df Residual	MS Residual	F	p
Particle size class	0.593557	6	382.0318	49	26.55442	14.38675	<0.0001
Median particle size (mm)	0.782495	8	25.66487	47	0.997333	25.73352	<0.0001
Fines (%)	0.859034	6	0.019585	49	0.000344	56.86086	<0.0001

Table 6. *Basic statistical properties from general regression model for stem wood chips (SW) predicting particle size parameters.*

Quality type	Adjusted R^2	df Model	MS Model	df Residual	MS Residual	F	p
Particle size class	0.626781	8	279.3465	49	21.54512	12.96565	<0.0001
Median particle size (mm)	0.704366	7	21.72253	50	1.064784	20.40086	<0.0001
Fines (%)	0.604959	4	0.000795	53	0.000035	22.82224	<0.0001

5 Discussion

5.1 The general validation model

The general validation model developed in this thesis originated from a need for a uniform and comparable method for testing measuring instruments for operational use in trading forest biomass fuels. When comparing studies on moisture content meters, the differences in experimental designs, statistical models, and diversity of results from the analytical methods made it difficult, although not impossible, to compare one meter with another (Järvinen, 2013; Leblon et al., 2013; Volpé, 2013; Fridh, 2012; Hultnas & Fernandez-Cano, 2012; López, 2012; Kullenberg et al., 2010; Roux et al., 2010; Jensen et al., 2006; Samuelsson et al., 2006; Andersson & Yngvesson, 1992; Blomqvist et al., 1986). Therefore, one objective of this thesis was to validate and compare instruments with different technical measuring principles.

For this, a statistically robust and reliable model was required, capable of providing comparable results for the accuracy and precision of the instruments regardless of the studied quality parameters concerning biomass materials, technical measurement principles, instrument models, or individual instruments. One of the strengths of the mixed model used in this thesis is its flexibility in modelling the random error and random effect variance components without requiring homogenous variance. For the samples tested, consisting of different biomass materials, levels of moisture content, and degrees of fraction size, it would have been impossible to meet the standard assumptions of a balanced fixed-factor GLM.

In the preparation of the samples, the initial moisture content was determined using a handheld moisture content meter, and the intention was to have at least five samples in each M_CL. In the statistical analysis, the division into M_CL was based on the reference moisture content after oven drying, and for some samples the drying process transferred them into another M_CL. This challenge, that there were sometimes four samples in one M_CL and six samples in the other after drying, was tackled by hypothesis testing using a Type III test, since it is independent of sample size. Also, the validation model has proved to introduce errors when the instrument's measurement values were compared with the reference method values within parameters. The reference measurements regarding samples were carried out in accordance with the standards for solid biofuels (ISO, 2016; ISO, 2015a; ISO, 2015b; CEN, 2010; CEN, 2009). These reference methods have, no matter how well they have been performed, a certain amount of errors, such as sample error for the ash and energy value determination, the oven drying procedure, and the precision of the scales used. These sources of error are included in deviations between instruments and reference methods, so the results for the instruments may be better than the results presented.

5.2 Sensor-based measuring instruments

The results for the instruments' ability to measure moisture content showed that the CAP had the lowest precision but was at the same level as other tested dielectric instruments (Volpé, 2013; Fridh, 2012; López, 2012; Jensen et al., 2006; Forsén & Tarvainen, 2000). The CAP also had the lowest accuracy, significantly underestimated the moisture content, and showed a high sensitivity to the moisture of the material, with major deviations when the moisture content was > 50%. The latter was expected as the manufacturer had calibrated the instrument for a moisture range of 12-40%. The attempt to obtain a calibration function gave a better accuracy but with unchanged precision. The calibration was based on the instrument's moisture content values from the lab test. It would be better to calibrate the instrument by creating completely new calibration curves based on the capacitance values recorded by the instrument (Volpé, 2013). However, the capacitance technology would benefit from calibration curves based on better described materials.

Because the technology is sensitive to density and moisture (Jensen *et al.*, 2006; Skaar, 1988), calibration would be needed for different materials and for different moisture content levels within the same material. The CAP instrument could then be suitable for smaller plants, with more specific demands, where the variation in the chip material is small, and with specific calibration curves for that unique chip material. One of the benefits of CAP is that it is a small and easy-to-use instrument. Up to 10-12 measurements can be quickly taken in a pile of chips, making it suitable for production measurement. In addition, the price of the instrument is about 1% of what the other instruments cost, which also makes it attractive for e.g. contractors or truck drivers to have a CAP with them in their vehicle.

MR had the highest repeatability (Figure 8), with slightly lower precision for the MR-C than for the MR-S. The MR-instruments also showed the highest accuracy in moisture content determination, independent of material. In the sensitivity test, the interaction (SORT x M_CL) showed a significant impact for MR-S but not for MR-C (Table 4). For three samples of logging residues in M_CL 5, it had a significant impact but not in any other materials and moisture content combinations. In theory, MR should be insensitive to the material (Järvinen, 2013; Sjöström, 2011) since it measures the electric current generated by the hydrogen atom spin after excitation. This unsensitivity for material is also is shown in these studies, except for the three samples of logging residues in M_CL 5.

MR has an advantage in that the calibration is done with plain water and does not require recalibration for different biomass materials. It measures any biomass material providing it is not magnetic. One disadvantage, however, is the small sample volume of 0.8 L which, in combination with the prerequisite of at least 20 g of water in the sample container, makes it difficult to measure the moisture content of coarse and dry chips. This problem can partly be solved by milling the sample before measuring but this is time consuming and therefore undesirable when developing fast measuring systems. Another option is to use a machine that accepts larger containers, but this would require a bigger and heavier instrument (Järvinen, 2013).

Overall, the NIR machine was well calibrated. Optical devices based on NIR reflectance have shown some promise in previous trials, in that they can be operated with high accuracy and with low sensitivity towards variable fuel types (Leblon *et al.*, 2013; Jensen *et al.*, 2006). The differences in precision between measurements on frozen and unfrozen material were very small, and there was no significant impact of the condition (frozen / unfrozen) on the DIFF_M between the NIR machine and the reference method. This indicates that the machine was well suited to measure both unfrozen and frozen material, as was also noted by Hans *et al.* (2013). It has previously been demonstrated that the NIR-instrument can also predict moisture content in frozen wood using PLS regression models (Thygesen & Lundqvist, 2000a; Thygesen & Lundqvist, 2000b), but now it has been shown to be an operational measuring instrument specially designed for biomass fuels.

The CXR results for the moisture measurements were uneven. The precision of repeated measurements was ± 2.3 pp and is somewhat less precise than for MR and NIR. The use of DXA technique for moisture measurements has been showed to be unsensitive for materials being frozen or not (Hultnas &

Fernandez-Cano, 2012; Kullenberg *et al.*, 2010). The CXR showed small difference between measurements of frozen or non-frozen material. Frozen stem wood chips in M_CL = 5 showed an underestimation of around - 2 pp, while unfrozen was overestimated by almost + 3 pp. All these M_CL 5 samples had a reference moisture content between 54 and 61%. These samples had to be measured using the scale for logging residue chips, since it was not possible to get complete data with the scale for stem wood chip. This indicates that the calibration space is too small or narrow for the specified material, and could possibly explain the differences in measurement values between frozen and non-frozen stem wood chips in M-CL 5. The analyses showed that the instrument is sensitive to SORT, M_CL and M_CON and had a systematic overestimation of the moisture content of M_CL 4 and 5. This reinforces the need of an adjusted calibration model since it is desirable that the accuracy is better than the test showed when moisture content is used as payment data.

The CXR managed to measure ash content with high accuracy and precision. In the instrument, the XRF-sensor are used to determine the ash content, at it has been previously shown that XRF is a suitable technology for detecting ash components (Thyrel et al., 2013). Based on how ash is measured in Sweden today, where samples from a supplier are mixed into a larger ‘monthly’ sample from which ash content is determined, the CXR accuracy and precision will be fully sufficient. Determination of ash content directly for each truck load allows better follow-up and control of fuel delivered, even if data is not used as a payment basis.

The CRX was consistent in measuring net calorific value, with a systematic underestimation between 0.33 to 0.53 MWh/ton, corresponding to an underestimation of 12-21% of the average net calorific value. The Swedish Timber Measurement Act (Anon., 2014) specifies a maximum deviation of 11-20% depending on the size of the delivery. This deviation also includes deviations of weight measurement error and sampling errors, so the instrument deviation should not exceed 5%. Since the instrument had a high precision this bias should be easy to calibrate for. The analyses established that the instrument is sensitive to the interaction ‘material’ and ‘moisture conditions’, which was the only significant interaction. After calibration, the instrument has a potential to determine net calorific value with an accuracy that enables the net calorific value determined to be used when the payment for the biomass delivered is calculated. This will remove some of the errors that may occur when the net calorific value is calculated from the moisture and ash content and the gross calorific values, as all these values are based on separate samples.

The CXR potential ability to predict particle size parameters is promising, but caution must be taken to interpret these results. The dataset was large enough to enable regression models to be made but these models will only be valid for the studied material. They cannot be used for determinations of the chip size parameters in a more general case. Since the models are "black-box" models, and not based on causal relationships between CXR data and specific material properties, a comprehensive validation process is of high importance. Continued studies are therefore necessary to determine the accuracy and precision of these models.

Sensor technologies that enable simultaneous measurement of many parameters, like CXR, will provide an advantage from a cost efficiency perspective. Any sensor based instrument that can be used to simultaneously determine the product and its quality characteristics from the same sample has a potential to be more cost efficient than the current methods used. Similar results should be possible using other techniques or combination thereof. One solution could be to combine image analysis and NIR-spectroscopy, there the image analysis provides information on the chip size parameters (Kuptz & Hartmann, 2015; Hartmann *et al.*, 2006) and NIR information on moisture (Fridh *et al.*, 2017; Lestander & Rhén, 2005), ash content (Thyrel *et al.*, 2013; Lestander & Rhén, 2005) and net calorific value (Lestander & Rhén, 2005).

As with CAP, the NIR and CXR must be calibrated for each unique material within a moisture gradient, but also for whether the material is frozen or not. The NIR instrument could self-detect the material in the 5L sample container, so the operator does not need to specify what material to measure. The measurement value for the material in shown on the instrument display, but gave no indication of how well it had estimated the measured value. The CXR, on the other hand, gave an indication of how well the instrument had managed to identify the sample, i.e. how good the sample was in accordance with the calibration space. For the CXR-instrument, a measuring scale had to be selected for the material in question to enable the correct calibration.

For the CXR the wettest samples of stem wood chips (M_CL 5), was not possible to measure using the scale for stem wood chips but successfully measured when using the scale for logging residues, as recommended by the manufacturer. This is a good example of what happens when the calibration space is too small or narrow for the specified material or, inversely, when a material or assortment spans such wide property variations that the properties overlap the assortment. It would be beneficial if the instruments could scan and identify the material presented for the sensor and select the appropriate scale based there upon, like the tested NIR instrument. For the measuring technologies studied in this thesis, more standardised descriptions of the traded forest fuels properties could be helpful for the initial construction of adequate calibration spaces.

Creating assortments just to facilitate the measurement is not realistic. But if these new assortments can provide benefits in the supply chain management or for the customer, more well-defined assortments could be created that are advantageous for measurement. However, to benefit from more detailed assortments, it is necessary to be able to confirm the assortments when the material is delivered. This can be provided by information gathered earlier in the supply chain and measurements of other characteristics than just moisture content at the time of delivery, or a combination thereof.

Of the studied instruments, only the CXR currently has the capacity to measure other characteristics than moisture content. With the possibilities to describe chip size distributions/proportion of fines shown in Paper V it should be possible to use an single instrument to discern and measure most assortments defined in Fridh et al. (2015). For the other studied instruments, assortments currently must be verified by supply chain information or measurements made by other techniques, such as sieving and ash content determination in lab.

5.3 Well-defined forest fuel properties

Forest and energy companies are calling for more efficient ordering and delivery of the right forest fuel to the right incineration plant, and this has become a priority issue for the entire sector (Iwarsson-Wide & Björheden, 2015). During the period 2013 to 2015, the value of the traded forest fuels has varied between 2.9 and 4.1 billion SEK at mill gate (Anon., 2016b). However, the products are not well defined and this is a great concern for all market actors in the sector.

In a joint project, Skogforsk, SDC and representatives of the sellers and buyers of forest fuel have developed a proposal for standardised forest fuel product descriptions (Fridh et al., 2015). Each fuel product is described based on whether they are comminuted or not, and distinguished further according to specified levels or ranges for ash and moisture content, fines and particle size class. The product description data will be gathered from measurement data, historical data, figures from experience, and prediction calculations, so that they can be followed in the production chain from stump to industry gate (Figure 12). Using well-described product characteristics throughout the chain of business can give the sector several advantages over the current situation:

- The buyer can place a more precise order, and receive fuels that match their specific needs.
- The supplier knows in detail what the buyer wants and can plan the supply activities accordingly.

- Both buyers and suppliers can get faster and more accurate forecasts and feedback on deliveries, in terms of both volumes and properties.
- Allows trading/switching of forest fuel between suppliers in order to reduce transport costs and environmental impact

For this to be effective, quality and quantities data should be collected continuously while the material is being handled or transferred in the supply chain (Sikanen et al., 2016).

Figure 12. Schematic overview of forest fuel supply chain and the collecting of characteristics data while the material is being handled or transferred.

Some of the major challenges when implementing this are: 1) to measure and determine the characteristics for production management at the different links in the production and supply chain; 2) to store and communicate the data and information to the next links in the supply chain; and 3) to be able to determine the product, and to measure the delivered amount and quality to enable calculation of the value of delivered forest fuel.

For the first challenge, further research is required on new technical solutions to measure the properties in flow or transhipment. The measurement must be cost-effective and take place during transhipment or chipping, and should be automated so that the load/unloading/chipping personnel do not lose production time due to measurement work. This will require a lot of R&D work to find smart and robust solutions.

For the second challenge, extensive development work is under way. SDC is developing a new timber trading system (SDC, 2017b) that will enable data collection across the entire supply chain, with the ability to handle large numbers of properties for each product or range. It is expected to be ready for operation in 2019.

The third challenge is perhaps the most important. If more properties than moisture content are to be determined, i.e. ash content, particle size and proportion of fine materials, the measurement of these must be more efficient and rational than current measurement methods. These parameters are currently measured through laboratory analyses (ISO, 2015a) or through manual methods such as screening and weighing (ISO, 2016). Any sensor-based instrument that can be used to simultaneously determine the product and its quality characteristics from the same sample has a potential to be more cost effective than the methods currently used. It may then be possible to allocate the measurement cost to several benefits or uses of the measurement data, i.e. for payment to supplier, for managing delivery/ supply flow, for controlling the combustion process, etc.

5.4 Sampling challenges

Regardless of the measuring technology, all the instruments measure samples, so it is very important that sampling is carried out systematically and carefully according to current instructions. One advantage of using an instrument is that it will conduct the measurement the same way regardless of where the measurement is done or who performs it. Another advantage is that all the tested instruments are non-destructive with respect to the samples, which is a great strength compared to the oven-drying method.

A major source of error in the measurement of moisture content with today's standard method is the sampling error (Vikinge & Gustavsson, 2016; Strömberg & Svärd, 2012). With a well-defined product specification for the fuel, together with more information about the fuel properties from earlier in the supply chain in combination with a sensor-based meter, the measurement of forest fuel would be more accurate and effective. However, this requires a different sampling and measurement instruction than the one available today.

According to the sampling instructions issued by SDC, six samples are to be taken, then mixed to form a general sample as the basis for moisture content determination (SDC, 2017a). In this procedure, much information about the fuel in the delivery is lost. Suppose a truck load of fuel enters a heating plant and is to be measured. Based on a more detailed product specification and preliminary information about the moisture content, e.g. from the chipping activity, an estimate of the moisture content of the material and its variation is obtained. With this information, the number of samples needed can then be determined for this specific truckload. To give an example, assume that six samples are required. All six samples are measured with an instrument and the mean and the variance are calculated. If the variance is greater than a certain given limit value, additional samples (e.g. two) are taken, measured and the mean and variance for all eight samples are calculated.

Table 7 shows a practical example of the number of samples needed to reach a certain degree of precision, based on the field study in Paper I. Stem wood chips were regarded as a relatively homogeneous material but Pile 2 exhibits very large variations within the pile and requires significantly more samples than the other piles to ensure measurement accuracy. By using a measuring instrument that quickly measures several samples and presents the mean and the variance, better information is obtained about the fuel's variation and renders a higher measurement quality. The ability to calculate the mean value and the variance of a desired number of measurements is a functionality missing in three of the four studied instruments. The CAP can calculate a continuous mean but not the variance. Calculation of mean and variance should not be difficult to achieve in an instrument, and it would increase the value of measurement data. The use of the variance to improve sampling will enable better measurement quality with an instrument than currently produced by the oven-drying method.

Table 7. *Moisture content variation within respective truck load (Pile) of stem wood chip from the field study in Paper I. The necessary samples (n) for the confidence interval (CI) include an instrument uncertainty of ± 2.5 percentage point (pp). Thirty samples from each pile were taken for reference analysis.*

	Pile 1	Pile 2	Pile 3	Pile 4	Pile 5
Number of reference samples	30	30	30	30	30
Moisture content range (%)	24.3 - 43.8	19.3 - 56.0	25.0 - 39.7	23.6 - 36.7	25.2 - 31.6
Mean moisture content (%)	31.8	39.5	32.0	28.8	28.9
Std. dev. of mean (pp)	4.4	7.4	3.1	2.6	1.8
Variance	19.36	54.76	9.61	6.76	3.24
Necessary samples n for CI within:					
± 2 pp n=	25	59	16	13	10
± 4 pp n=	7	15	4	4	3
± 6 pp n=	3	7	2	2	2
± 8 pp n=	2	4	1	1	1

Although the oven-drying method is the reference, there are many contributors to the source of error. In a study (SCAN, 1994), 30 samples were taken from the same pile, split into 3 equal-sized sub-samples, and sent to three different laboratories for dry matter determination. This resulted in a coefficient of variation (cv) of 2 to 3% within labs, and a cv of 3% across labs. The overall dry matter content of 58.3% in that test equates to a standard deviation of 1.7 pp in the moisture content estimates of the three labs.

During oven drying of samples, there is always a risk that some volatile compounds are lost from the samples during the drying process. Depending on the material, losses of volatile compounds during drying cause differences in moisture content of more than one percentage point if the drying is carried out at the standard 105 ˚C instead of 80 ˚C (Samuelsson et al., 2006). The temperature inside the oven is very important for the drying time (the same material can take 16 h at 120 ˚C, but 28 h at 85 ˚C), and there may be considerable differences (10-15 ˚C) in temperature at different places inside the oven, often higher than the temperature the oven is set to (Björklund & Fryk, 1989).

The instructions stipulate that the samples should be dried and then weighed, dried an additional hour and weighed again to determine whether they are completely dry. During the many study visits to heating plants and measuring stations during the work with this thesis, it was noted that this rarely happens in the operational measurement procedure. Instead, a standard time is used for the samples in the oven, varying from 20 to 36 hours depending on the measurement site visited (arbitrary based decision). There were also differences in the practical use of the oven. At some measuring stations, the entire oven was filled with samples before starting the drying process. Others continuously added and took out samples from the oven, which could result in longer drying time. Björklund and Fryk (1989) found that the drying time varies significantly depending on the material, and that it cannot be ruled out that it also is affected by continuous insertion and removal of samples to and from the oven. In theory, the oven-drying method is precise if the samples are dried to constant weight, but the variations in operational procedure are likely to induce errors in the moisture content determination.

One way to minimise or eliminate the sampling errors is to measure the entire load or much of it, e.g. by measuring in a flow or measuring the entire or much of the truck load when on arrival at the heating plant. Several methods are available for flow measurement of chips, such as NIR, X-ray, microwave and radio frequency (APOS, 2017; Inadco, 2017; Inray, 2017; Mantex, 2017a; Senfit, 2017), but they are intended for process management and need to be installed in feeding systems to boilers, on conveyor belts or screw feeders. An application for these methods when measuring incoming trucks would require extensive installations and space at the heating plant storage facilities, which could only be possible for a few of the largest heating/power plants. Attempts have been made to measure the moisture content before unloading the trucks using radar technology. For small containers the results showed that an accuracy of 3-4 pp could be obtained (Ottosson *et al.*, 2016).

6 Conclusions

Sensor-based measuring equipment that can detect and determine more fuel properties during the same measurement, or other properties such as chemical composition and fibre qualities, will have a major competitive advantage. The strength of these technologies is the fast determination of fuel properties and their variance and the possibility to use this to optimize sampling. Through well-defined assortments based on customer requirements for fuel properties, together with advance information about the fuel from automated measurements along the production chain, sampling and thereby measurement accuracy can be improved. It also enables better conditions for the calibration and precision of the sensor-based instruments.

- Sampling and handling of samples is extremely important for a thorough estimation of moisture content in a truck load of chips, regardless of whether the oven method or any of the studied instruments are used.
- A sensor-based measuring instrument provides a more consistent measurement method than the oven-drying method.
- The three instruments, MR, NIR and CXR, are well suited for payment-based measurement at industry and larger storage terminals, but not for field use.
- The CAP instrument is well suited for field use and for smaller reception locations, and can be used for payment-based measurement under certain limited conditions.
- With preliminary information about the properties of the fuel material from earlier stages in the production chain, a more appropriate sampling and measurement method can be selected.

7 Suggested further research

New measurement technology must be developed for the continued efficient management and trade of forest biomass for fuel or other uses.

In the agricultural sector, NIR technology is used commercially to measure moisture content in grain directly in the combine harvester (Paul et al., 2008; Roberts et al., 2004), at the exhaust when loading the grain to the side wagon. Suppose it is possible to mount sensor-based measurement equipment in the chip feeder directly after the chip drum, where chips are fed to the outlet. If a truck is loaded directly from the chipper, the moisture content can be determined for each container on the truck. With this information before delivery an appropriate level of sampling can be determined for measurement at the plant, which could improve the quality of the measurement for individual truck loads.

The pilot study of radar technology showed promising results. If the accuracy of 3-4 pp could be obtained when a when the truck slowly passes two radar antennas, 50-70% of the total truck load of chips could in theory be measured. This would mean a significant improvement in sampling. Furthermore, the technology may be adapted to other applications e.g. to mount it in a chipper or in a bucket on a wheel loader. These two applications would then be beneficial for measuring in the production flow when loading chips, to provide better advance information and to manage production.

Another challenge is to measure moisture content of logging residues, thinning materials and roundwood (non-comminuted fuels). There are currently no effective methods, even if there are one or two measuring stations in Sweden that have a mechanised sampler (from the 1980s), using a drill to collect material (Björklund, 2014). Sampling using chainsaw has been studied, where sawdust samples are collected by cutting incisions into the materials accessible from the outside of a truck load (Nylinder & Fryk, 2012).

To meet these challenges, continued research and development of measurement technology solutions, forest fuel product descriptions, and data and information management throughout the forest fuel supply chain is required.

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Popular science summary

The use of primary forest fuels increased rapidly during the first decade of the twentieth century. As these assortments were not included in the 1966 timber measurement act, which only regulated the measurement of conifer sawlogs and pulpwood, it was considered that there could be a risk that the measurement was not carried out in a credible, similar and quality assured manner throughout the country. Therefore, the Swedish Forest Agency 2008 initiated the work to revise the provisions of the Act. This led to a revision of the legal text, and a new act in 2015, that include all assortments. The new law has led to increased demands for accuracy and precision in the measurement of forest fuels. As the most common commercial measure for forest fuel is the net calorific value, the weight, moisture content and ash content of the delivered fuel must be measured with acceptable precision and accuracy. At present, the moisture content of a chip sample is determined by the oven-drying method, which means that the sample is weighed, then dried at 105 °C until constant weight is reached and then weighs again. The moisture content is then calculated by dividing the weight loss by weight before drying. The method is slow and a faster and more cost-effective method of measuring the moisture content that can be used for remuneration measurement and production control in the supply chain is necessary.

The purpose of the work was: a) to develop a statistically robust validation method for precision and accuracy in moisture content measurement, b) to validate the precision and accuracy of moisture content measurement in chips for measuring instruments using electrical capacitance, magnetic resonance (MR), near infrared (NIR) spectroscopy or X-ray technique to determine the moisture content, and c) to evaluate the possibilities of estimating other parameters using X-ray techniques.

The results show that, apart from the capacitance meter, the studied instruments had a precision where more than 95% of the measurements within ± 2.5 percentage points from the mean of repeated measurements on the same sample. There were large variations in accuracy between the instruments studied and the accuracy was also influenced by the moisture content of the material, the type of material, and whether the material was frozen or not. The MR instrument provided the most accurate measurement results of the measured meters, and its precision was not affected by the type of material. Since the accuracy is a systematic deviation, the deviations can be corrected by a calibration of the instruments. In addition to estimating moisture content, the X-ray instrument can also estimate ash content and effective heat value for the chips. The analysis of the possibilities of using X-ray technology to estimate more quality parameters enabled it to estimate both the fine fraction and the median particle size of the sample with relatively high security.

The capacitance instrument, was the only one of the tested instruments that was easy to transport and use in fields, and therefore the only one adapted for measurements intended for production management. The other instruments are intended for use at measuring stations at larger terminals or reception sites at heating or combined heat and power plants. The precision of the instruments tested, in addition to magnetic resonance, was influenced by the type of material. A more accurate division of the chips in the fuel assortments would facilitate the calibration of these instruments. A clearer division of the assortments based on their characteristics such as moisture content, particle size, fine fraction etc. would also benefit the fuel customers who can use this information to increase the efficiency of their facilities. But in order to achieve these benefits, it must be possible to check that the delivered fuel material complies with the delivered assortment. This could be done with sensors, and in some cases, it can be done with the same instrument that measures the moisture content. For example, the studied X-ray instrument can determine ash content and net calorific value and the results indicate that it is possible to estimate fine fraction and median particle size. This should suffice to verify that the delivered fuel material is the expected assortment. Both the oven-drying method and the instruments studied determine the moisture content of samples. This means that proper sampling is critical to a moisture determination for a forest fuel supply, and assumes that a sufficient number of samples are taken to minimize sampling errors. Previous studies have shown that sampling errors cause a large part of the total error in the moisture determination of a delivery. A measurement method that determines the moisture content in such a short time that the customer can use the information to improve the combustion process can in many cases be a more cost effective alternative than the oven method, especially if the instrument can measure more quality parameters.

Populärvetenskaplig sammanfattning

Användningen av primära skogsbränslen ökade snabbt under tjugohundratalets första decennium. Då dessa sortiment inte omfattades av 1966 års virkesmätningslag, som bara reglerade mätning av barrsågtimmer och massaved, ansågs det att det kunde finnas en risk att mätningen inte genomfördes på ett trovärdigt, likartat och kvalitetssäkrat sätt i hela landet. Därför inledde Skogsstyrelsen 2008 arbetet med att revidera föreskrifterna till lagen. Detta ledde till att även lagtexten reviderades och från och med 2015 gäller en ny virkesmätningslag som inkluderar alla sortiment. Den nya lagen har lett till ökade krav på noggrannhet och precision i mätningen av skogsbränslen. Då det vanligaste handelsmåttet för skogsbränslen är det s.k. effektiva värmevärdet måste vikten, fukthalten och askhalten hos det levererade bränslet mätas med acceptabel precision och noggrannhet. I nuläget bestäms fukthalten för ett flisprov med den s.k. ugnsmetoden, vilket innebär att provet vägs, torkas i 105°C till dess att vikten slutar minska, och då vägs igen. Fukthalten beräknas sedan genom att viktminskningen delas med vikten före torkning. Metoden är långsam och en snabbare och mer kostnadseffektiv metod för att mäta fukthalten som kan användas för vederlagsmätning och produktionsstyrning i försörjningskedjan är nödvändig.

Syftet med arbetet var a) att utveckla en statistiskt robust valideringsmetod för precision och noggrannhet vid fukthaltsmätning, b) att validera precisionen och noggrannheten vid fukthaltsmätning i flis med mätinstrument som använder elektrisk kapacitans, magnetresonans (MR), Nära infraröd, (NIR)-spektroskopi eller röntgenteknik för att bestämma fukthalten, och c) att utvärdera möjligheterna att skatta andra parametrar med hjälp av röntgenteknik.

Resultaten visar att frånsett kapacitansmätaren så hade de studerade instrumenten en precision där mer än 95% av mätningarna återfinns inom ± 2,5%-enheter från medelvärdet vid upprepade mätningar på samma prov. Det fanns stora variationer i noggrannhet mellan de studerade instrumenten och noggrannheten påverkades även av fukthalten på materialet, typen av material, och om materialet var fruset eller ej. MR-instrumentet gav de mest noggranna mätresultaten av de studerade mätarna, och dess precision påverkades inte av typen av material. Då noggrannheten är en systematisk avvikelse kan avvikelserna korrigeras med en kalibrering av instrumenten. Förutom att skatta fukthalten kan röntgeninstrumentet även skatta askhalt och effektivt värmevärde för flisen. Analysen av möjligheterna att använda röntgenteknik för att skatta fler kvalitetsparametrar gav att det är möjligt att skatta både finfraktionsandelen och medianpartikelstorleken i provet med förhållandevis stor säkerhet.

Av de testade instrumenten är endast kapacitansinstrumentet lätt att transportera och använda i fält, och därmed det enda som är anpassat för mätningar avsedda för produktionsstyrning. De övriga instrumenten är avsedda att användas på mätstationer vid större terminaler eller mottagningsplatser vid värme- eller kraftvärmeverk. Precisionen hos de testade instrumenten, förutom magnetresonans, påverkades av typen av material. En mer noggrann uppdelning av flisen i bränslesortimentet skulle underlätta kalibreringen av dessa instrument. En tydligare uppdelning av sortimenten utifrån deras egenskaper som t.ex. fukthalt, partikelstorlek, finfraktionsandel etc. skulle dessutom gagna bränslekunderna som kan använda informationen för att öka effektiviteten i sina anläggningar. Men för att nå dessa fördelar måste det gå att kontrollera att det levererade bränsle materialet överensstämmer med det levererade sortimentet. Detta skulle kunna göras med sensorer, och i en del fall går det att göra med samma instrument som mäter fukthalten. Exempelvis klarar det studerade röntgeninstrumentet att bestämma askhalt och effektivt bränslevärde och resultaten visar på att det är möjligt att skatta finfraktionsandel och medianpartikelstorlek. Detta borde räcka för att verifiera att sortimentet är det förväntade. Såväl ugnsmetoden som de studerade instrumenten fastställer fukthalten för prover. Detta gör att en korrekt sampling är avgörande för en fukthaltsbestämning för en skogsbränsleleverans, och förutsätter att en tillräcklig mängd prover tas för att minimera samplingsfelet. Tidigare studier har visat att samplingsfel orsakar en stor del av det totala felet vid fukthaltsbestämningen av en leverans. En mätmetod som bestämmer fukthalten på så kort tid att kunden kan utnyttja informationen för att förbättra förbränningsprocessen kan i många fall vara ett mer kostnadseffektivt alternativ än ugnsmetoden, särskilt om instrumentet kan mäta fler kvalitetsparametrar.

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