

Determination of Moisture Content in Pine Wood Chips with Dual Energy X-ray Absorptiometry



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Abstract

Determination of moisture content (mc) in pine wood chips is of interest to the pulp and paper industry. The gravimetric method used nowadays is reliable, but it is slow and labours. The aim of this paper was to evaluate the accuracy and precision when determining mc in pine wood chips with dual energy x-ray absorptiometry. Also to see if the same algorithm could be used for both spruce and pine chips. This new method was evaluated and compared with the results achieved with the gravimetric method that was used as a reference. In this study 70 samples of pine wood chips ranging different mc were used. Mc was determined by both methods and results were analysed by regression analysis. This study showed positive results; standard error of estimate was 2.01%, standard deviation was 0.45%, and R^2 value was 98%. These results were similar to an earlier study conducted with spruce wood chips. However the correlation between both methods lacked of accuracy when the mc was low. These results showed that it is needed to use a separate algorithm for pine wood chips.

Keywords: moisture content, dual energy x-ray absorptiometry, pine wood chips

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1. Introduction

Successive improvements in techniques of measuring moisture content (mc) are needed for processes of wood to become more efficient. Processing natural resources more efficiently involves that only the necessary raw materials for production will be used. Therefore, at the same time that final forest products are obtained, energy is saved and pollution decreases. The study of determination of mc in wood is of importance for several forestry sectors such as paper industry and bio-energy. Today, the increasing trade in biofuels, both national and international, leads to the replacement of fossil fuels by renewable energy sources. Research on this field is very extensive nowadays, and the knowledge about mc is necessary for performing wood trade and processing forest products. Several research activities have been carried out to look for reliable methods to determine mc in wood chips (Nyström & Dahlquist, 2004). So far, current methods are not very reliable or the measurement process requires a long time.

1.1 Problem

Determination of mc is relevant to the forest industry. Specially, measurement of mc in pine wood chips is of interest for the chemical pulp and paper production. Mc is a quality requirement in the wood chips supply to the pulp industry; it is also a control parameter within the pulping process. Prize setting of wood chips deliveries to the mill depends on mc to a great extent. Truck loads of wood chips are purchased by weight, which is highly influenced by the water contained in the wood. It is of interest for the pulp industry and for the producers of wood chips to have a quick, representative, and reliable method to determine mc in wood chips. Mc works as a process parameter that needs to be controlled for pulp processing. When adding liquors to the digester in the chemical processes, concentration of chemicals must be established, and then, it is necessary to accurately determine the mc of wood chips. The most desired measuring system would be an on-line determination of mc. In this way, it could be incorporated to the whole system, and mc parameter would be controlled in real time.

Mc determination can be of interest also for energy plants and board-mills, in order to establish an even mc in the incoming flow of wood chips to the process. In energy plants an even mc in the flow of wood chips into the combustion chamber is needed to achieve efficiency. Controlling the mc of the incoming flow would allow to make the necessary adjustments of the combustion process.

The method to determine mc used nowadays is the gravimetric method; it quantifies moisture by establishing the difference in weight between wet wood and totally dry wood. This method needs long time (24 hours), and space to dry enough number of samples to get a representative result. The gravimetric method has proved to be reliable; however, the long time required and the labour needed are great disadvantages. Moreover, by this method it is impossible to get on-line measuring of mc.

NIR, radio frequency and dual energy x-ray absorptiometry techniques are apparently suitable to solve these problems, and bring to the forest industry a method that can determine mc quickly and with high accuracy. Moreover, it would be necessary that the method could also determine mc in frozen or semi-frozen samples of wood chips. The three mentioned methods are in an early stage and need further development to achieve these goals. (Hultnäs, unpublished).

1.2 Aim

The objective of this research was to verify the prototype based on dual energy x-ray absorptiometry developed by Mantex as potential technique to determine mc in pine wood chips. This study has been done without performing any new calibration to the prototype. The software of the prototype was calibrated for spruce wood chips, for the study of Hulnäs *et al.*, 2009. Verification of the method for pine wood chips was done comparing results with a reference method. The gravimetric method was the reference method used in this study, since it is the method commonly used today to determine mc in wood. Results were analysed with regression analysis to prove how well correlation was between both methods.

1.4 Abbreviations, definitions and units

Abbreviations and units

mc	Moisture content
SD	Standard Deviation
SE	Standard Error of Estimate
St.	Standardized
R	Correlation coefficient
R ²	Coefficient of determination (R-squared)
Obs.	Observation
DXA	Dual x-ray absorptiometry
FSP	Fibre Saturation Point
GROT	“Grenar och toppar” (branches and tops)
VMF	VirkesMätning Föreningen (Swedish Wood Measuring Association)
SLU	Swedish University of Agricultural Sciences. “Sveriges lantbruksuniversitet”
°C	Celsius degrees
keVp	Kilo electron volt potential

Definitions

In this paper terminology used and their definitions are explained as follow:

Absorptiometry: is a measurement of absorption, of radiation. (MediLexicon, 2009)

Algorithm: is a procedure that provides a solution to a problem, and consists of a set of unambiguous rules which specify a finite sequence of operations. Each step of an algorithm must be precisely defined and the necessary actions must be rigorously specified for each case. An algorithm should always arrive at a problem solution after a finite and reasonable number of steps. An algorithm that satisfies these requirements can be programmed as software for a digital computer. (Longley *et al.*, 2005).

Attenuation: is a general term that refers to any reduction in the strength of a signal. Attenuation occurs with any type of signal, whether digital or analogue. Sometimes called loss, attenuation is a natural consequence of signal transmission over long distances. (Networking-specific information resource for enterprise IT professionals).

Bark content: is the proportion of bark in wood chips; some bark remains from the debarking process and bark is found together with wood chips after chipping. Bark content is found as

small pieces of bark attached to wood chips or as loose pieces of pure bark; proportions of bark content in wood chips vary from slightly nothing to an almost complete chip of bark.

Dual x-ray absorptiometry (DXA): is an imaging test that first applications were to measure bone density (the amount of bone mineral contained in a certain volume of bone) by passing x-rays with two different energy levels through the bone. (National Cancer Institute).

Gravimetric method: consist of weighing the wood at the moment needed to know its mc, dry this wood at 105 ± 2 °C for at least 24 hours until constant weight is achieved and then weigh it again. It is the reference method used in this study to compare the results. The gravimetric method follows the Swedish Standard 18 71 70. (Swedish Standard, 1997).

GROT: is a Swedish term that refers approximately to forest residues. GROT is a by-product of clear-cutting, composed of branches, tree tops and small trees. GROT cannot be used for pulp or timber production. (Löfstedt, 1996).

Mantex method: uses a new technology as an alternative for fast determination of mc. It is based on DXA technique. Two x-ray beams are produced and are partly absorbed by the material; the software provides the percentage of mc of the sample.

Percentage of mc: is the amount of water in wood. The mc determined by the gravimetric method was calculated with the formula (1).

$$mc(\%) = \frac{(W_1 - W_0)}{W_1} * 100 \quad (1)$$

mc (%) = Moisture content in percentage.

W_1 = Weight of the sample wet.

W_0 = Weight of the sample dried.

Sawmill wood chips: are wood-chips coming from sawmills; they consist to a great extent on sapwood chips. They contain bark in different proportions.

2 Background

2.1 The Swedish wood market

Forests provide wood to the forest industry. In a first overview of the wood market, wood raw material is delivered to sawmills, pulp- and papermills, boardmills and energy plants for processing in order to obtain the wanted products. (Bjurulf, 2006).

However, since there are purchases of raw material to each of the wood processes, wood can be traded several times, and several products and by-products are obtained by the companies and sold to other companies. Usually, sawmills, pulp-mills, board-mills and energy plants receive wood from many suppliers, and have a large number of costumers. Industries receive raw material, products and by-products from other wood industries. It can be that a harvesting site produces raw material directly to a sawmill, a pulp and paper-mill and an energy plant; from the sawmill it is obtained a product, timber, and by-products of the sawmill are sold to the pulp industry and the energy plant. The pulp-mill may also produce rest products for the energy plant. Figure 2.1. This fact creates a network increasing the complexity of the wood market. (Bjurulf, 2006).

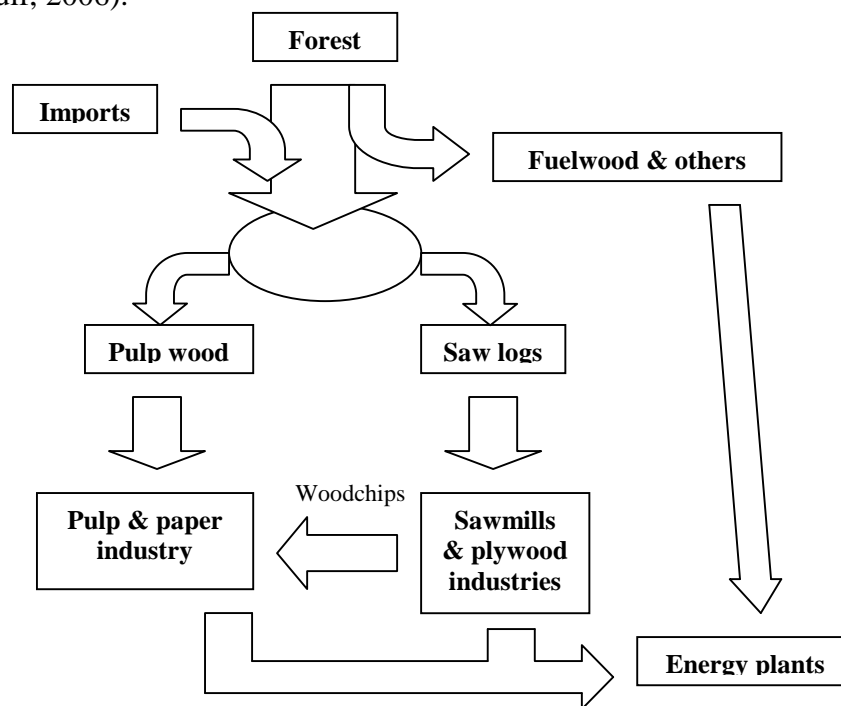


Figure 2.1. Flow chart of wood (Bjurulf, 2006; Skogsindustrierna, 2008).

Classification of wood starts already when logs are obtained from harvesting in the forest. Logs are sorted in special timber, logs for sawmills (first class, second class or low quality), logs for pulp-mill which are not good enough to be sawn (different tree species, high quality logs and low quality logs) and logs for energy production, moreover, harvesting waste for energy production. The wood market flow of different products starts since harvesting in the forest, and continues in different forms. As an example sawmill products are shown in Table 2.1. Initially sawmills produced mainly sawn-wood, however in recent times by-products have become widely used and acquired a great market value. (Bjurulf, 2006).

Table 2.1. Yield from sawmills in Sweden 2000 (Staland, 2002; cited in Bjurulf, 2006)

Wood products	Percentage
Sawn-wood	47
Wood-chips	34
Sawdust	10
Slabs	1
Rest	8

In the wood market every product has a use. Roundwood and woodchips are the main products coming from the forest, but harvesting waste for energy production has increased its market value nowadays.

Consumption of forest raw materials (including roundwood and other products) in Sweden was 86.6 million of m³ in 2006. In order to provide more information, the following figures are presented in Table 2.2. The figures are wood volumes in cubic meters excluding bark. Sawmill production consumed 37.8 million m³, but also produced 12.2 million m³ of chips and sawdust for consumption in the pulp and wood board industry. (Swedish Forest Agency, 2008).

Table 2.2. Consumption of forest raw materials in Sweden in 2006. (Swedish Forest Agency, 2008)

Production	Consumption (million m ³ excluding bark)
Wood board	1.2
Pulp and paper	47.6
Sawmill	37.8

Industrial roundwood consumption in 2006 is found in percentages classified into species in Table 2.3. Pine occupies the second position of importance in the consumption ranking. (Swedish Forest Agency, 2008).

Table 2.3. Roundwood consumption in percentage for tree species in Sweden (Swedish Forest Agency, 2008)

Wood Species	Percentage
Spruce	53
Pine	36
Birch	9
Other broadleaves	3

The energy supplied in Sweden from biofuels was 19% of the total energy supplied in 2006. Biofuels are primarily wood fuels: felling waste, industry waste, *Salix sp.* plantations, peat, etc. This energy that was used as it is shown in Table 2.4. The use of wood fuels is increasing for district heating and for electricity production. In the forest industries the use of wood fuels has mainly the same purposes, heating and electricity production. (Swedish Forest Agency, 2008).

Table 2.4. Uses of the energy coming from wood fuels (Swedish Forest Agency, 2008)

Use	Energy from wood fuels
In the Industry	54%
For district heating	37%
Others	9%

2.2 Characteristics of tree and wood

A brief description of the tree provides a useful basis for this study. Tree is generally defined as a woody plant of 5 m or more in height, with a single trunk rather than several stems. Trees are divided into two categories: hardwoods and softwoods. Hardwoods bear broad leaves, are generally deciduous, and their fruiting bodies produce acorns, pods and others. Softwoods are commonly evergreen, with needlelike leaves, and usually bearing scaly cones so that they are often referred as conifers. Softwoods occur naturally in the Northern Hemisphere and sporadically in the Southern Hemisphere. Wood of these two categories also differs, structurally and morphologically. (Haygreen & Bowyer, 1996).

Trees manufacture its own food by the photosynthesis process. Roots take water from the soil, and leaves take carbon dioxide from the atmosphere and collect the energy from the sun. This process produces the appreciated raw material, wood. Wood is produced by the cambium, an inner layer in the stem. Wood is specifically xylem; cambium produces xylem inwards to the stem and phloem outwards. Xylem differentiates in sapwood (living cells) and heartwood (dead cells). Sapwood transports the sap up and down the stem, roots and branches. Sap is mineral-rich water with sugars and other substances as growth regulators. (Haygreen & Bowyer, 1996).

Wood is composed by organic constituents, mineral matter and water. Within organic constituents, their main components are the polymers: cellulose, hemicelluloses and lignin. Their elemental composition is shown in Table 2.5. Proportions of cellulose, hemicelluloses and lignin vary between softwoods and hardwoods as it is shown in Table 2.6. These polymers form the different cells or fibres that constitute wood. The wood cell has a hollow centre called lumen and the walls of the cell are formed by different layers giving resistance. (Haygreen & Bowyer, 1996; Smook, 1990).

Table 2.5. Elemental composition of the organic constituents in wood (Gómez, 2007/2008)

Element	Percentage of dry weight
Carbon	50
Oxygen	43
Hydrogen	6
Nitrogen	<1
Mineral salts	<1

Table 2.6. Difference in composition of organic constituents between hardwood and softwood; percentages of dry weight (Haygreen & Bowyer, 1996)

Type	Cellulose (%)	Hemicelluloses (%)	Lignin (%)
Hardwood	40-44	15-35	18-25
Softwood	40-44	20-32	25-35

Water is a prerequisite for life, and it is found mostly in the living parts of the tree. Water is of great proportion in green wood, it commonly contributes with half of the total weight. In wood, water can be found in its three different states. For each state of water, its structure changes and so its properties. (Haygreen & Bowyer, 1996).

Water in wood is stored in the cell wall and in the cell lumen. The water of the cell lumen is known as sap and may contain dissolved substances. The water contained in the lumen usually is found as liquid and saturated water vapour. This water is the first removed when wood is drying and this is why it is referred to as free water. The water in the cell wall is referred to as bound water and it is linked by physicochemical forces. Water of the cell wall is found in the amorphous regions of the wood structure. Some molecules are more strongly bounded than others. (Haygreen & Bowyer, 1996).

When wood is drying what first occurs is that liquid water leaves the lumen. Once all the liquid water has been removed and the cell wall is still saturated with water, wood reaches the point termed fibre saturation point (FSP). This point is of importance because every change of mc below this point will affect wood properties in a considerable way. When continue drying below the FSP, water is removed from the cell wall and remaining water is the more tightly bounded. (Haygreen & Bowyer, 1996).

2.3 Scots pine (*Pinus sylvestris* L.)

Scots Pine (*Pinus sylvestris* L.) is one of the most widely distributed pines in the world, with many races and varieties recognized. It is native to Europe (from Norway to Spain) and to parts of Asia. In Europe and Asia, Scots pine forms a boreal forest type with Norway Spruce (*Picea abies* (L.)H. Karst). It is introduced in many areas in the United States and Canada, and is naturalized in the Northeast and in the Great Lakes states (Sullivan, 1993). Figure 2.2.



Figure 2.2. Scots pine forest, Gotska Sandön, in the Baltic Sea, north from Gotland (Håkan Svensson, 2000).

It is an evergreen tree up to 35 m high or more, with a trunk diameter up to 0.5 m. The canopy of young trees has conical shape, becoming flat-topped when getting older. Scots pine has a medium growth rate. The seed cones are globosely, pointed ovoid-conic, grey-green to yellow-brown at maturity, 3-7.5 cm in length, reaching full size in their second year. The cone scales have a flat to pyramidal apophysis, with a small prickle on the umbo. Needles are gathered in clusters of two, they are waxy bluish green, 4-6 cm long, twisted, sharp-pointed, and more or less significantly rigid and thick. They persist three years in the tree. The bark is

very characteristic in this pine, it is thicker scaly dark grey-brown on the lower trunk; and it is thinner, flaky and orange on the upper trunk and branches. The wood is mainly used for pulp and sawn timber products. (Ginés, 2002; Ruiz de la Torre, 1979).

Scots Pine has light yellow coloured sapwood, while heartwood is between pink and reddish brown. The colour of the knots varies between dark brown to blackish. Growth rings are easy to distinguish, as there is a clear difference between spring wood (yellowish white) and autumn wood (brown). The width of the growth rings varies between 1-3 mm to 7-8 mm. The resin ducts are common, not easy noticeable, of little or medium size, and usually isolated or in couples. In a transverse view are recognized as white points, and in a longitudinal view they are recognized as thin and short brown lines. The grain of the wood is classified between fine and medium. The smell of its resin is intense in raw wood and stands for some time after drying the wood. (Vignote, 2008).

For the pulp industry the types of cells that the species has are of great importance. Types of cells from softwoods fit better to the requirements of the technology applied to obtain the best product. Softwood is composed by fibres and parenchyma cells. The most important cells in pulp production are fibres; in softwood fibres are tracheids. Fibre tracheids, more properly called, are long, tapered and usually thick-walled cells of hard-wood xylem. Softwood tracheids are nearly rectangular in the cross section. With a length of 2-4.5 mm, they are considerably longer than hardwood tracheids. This fact makes them preferred for pulp production (mechanical pulp, Kraft pulp and unbleached paper products). Parenchyma generally does not produce any problem in pulp industry, but it can cause some problems when it contains silica crystal because it is abrasive. Then the pulp produces wearing in several processing machines. Table 2.7 shows the percentages of the different kind of cells contained in Scots pine and Norway spruce. (Haygreen & Bowyer, 1996; Smook, 1990).

Table 2.7. Cell types in percentage in Scots pine and Norway spruce (Ejderby, 1975; cited in Bjurulf, 2006)

	Scots pine <i>(Pinus sylvestris L.)</i>	Norway spruce <i>(Picea abies L.)</i>
Fibers, tracheids	93	95
Parenchym cells	7	5

Wood compounds in Scots pine and Norway spruce are very similar. The most marked differences between both species are in total extractives and in residual constituents. Scots pine has more total extractives and lower residual constituents than spruce. The three main components of pine and spruce woods are the polymers: cellulose, hemicelluloses and lignin. Table 2.8.

Table 2.8. Wood compounds in percentage for Scots pine and Norway spruce (Sjöström, 1981; cited in Bjurulf, 2006)

	Scots pine (<i>Pinus sylvestris</i> L.) (%)	Norway spruce (<i>Picea abies</i> L.) (%)
Cellulose	40.0	41.7
Hemicelluloses	24.9	24.9
Lignin	27.7	27.4
Other polysaccharides	3.6	3.4
Total extractives	3.5	1.7
Residual constituents	0.3	0.9

2.4 Chip properties

Requirements of wood chips properties differ between the different industries. Pulp industries set the highest requirements, and wood fuels accept lowest quality and most variation. The most common properties controlled for pulp production are: contaminations, mc, bark content, chip dimensions, and discoloration (Bjurulf, 2006). Sweden, Finland and Norway use standards to normalize analysis within the areas of wood chips. These standards are developed by the SCAN-test organization (Scandinavian Pulp, Paper and Board Testing Committee). There are eleven standards and the most used are the ones regulating: dry matter content, size distribution, sampling, and bark content. (Hedenberg, 1999; cited in Bjurulf, 2006).

The pulp industry needs homogenized mc in the flow of wood chips into the process. It is the same case for bio-energy and board industries. Mc changes throughout the year depending on climate and other conditions. (Smook, 1990).

Size and geometry of wood chips are of great importance to the pulp industry. It determinates a proper cooking in the digester, avoiding a low quality production. Over sized wood chips need to be rechipped; an adequate chip in size avoids losing time and energy. For chemical pulping, an ideal size and geometry of a chip consist of approximate the following measures: 10 to 30 mm in length, 20 mm in width, and 2 to 5 mm in height. When cooking, height and width helps chemicals to reach the centre of the chip through two processes, osmosis and capillary action. Chemicals diffusion through osmosis is in width direction, and capillary action occurs in height direction. When wood chips are too big, they result in uncooked chips that need to be removed from the pulp; this occurs due to the impossibility of the chemicals to reach the centre of the chip. Uncooked chips occur also when a too low concentration of chemicals is added due to miss-calculation of the mc, and bark content. On the other hand when wood chips are too small, they result overcooked. This means that chemicals destroy all the lignin and also attack cellulose and hemicelluloses destroying fibre length and results in reduced pulp-yield. In semi-chemical pulping in general wood chips size must decrease since chemical treatment within the process is short in time and lighter (less aggressive). (Smook, 1990).

Bark content contained in the wood chips produces several problems for pulp industry. It introduces several contaminants (i.e. stones, sand, plastic, metals, etc) and it makes the pulp hard to bleach. (Bjurulf, 2006).

2.5 Importance of measuring mc for the forest industries

Pulp and paper industry, board industry and bio-energy industry need to control mc in wood chips as successfully as possible. Information about mc helps to establish an adequate price when trading in wood chips, to achieve a uniform mc in the incoming flow of wood chips to the mill and to calculate an adequate supply of chemicals to the digester.

Importance of mc for the pulp industry

The supply of wood chips to the pulp industries represents around 25% of the total wood supply (Skogsindustrierna, 2008). Payment for the wood chips is usually based on the dry weight of the fibres, and then determination of mc becomes important to differentiate water from wood and not to pay for the water as purchasing wood. Size, mc and origin of wood chips are carefully controlled. Sorting must be strictly controlled and categories not mixed; each category requires specific process settings. In the wood yard of the mill, wood chips are sorted out by origin and species; so the incoming flow of wood chips to the pulping process tries to achieve similar mc. Roundwood is not paid by dry weight, but after chipping, an uneven mc in the flow of wood chips to the pulping process causes impregnation problems. (Smook, 1990; Bjurulf, 2006).

Coniferous are used for mechanical, chemical and semi-chemical pulping. Table 2.9 provides an idea of the importance of each pulping process. It shows the raw materials used in the paper industry; pulp coming from the pulp industry and from recovered paper, and also fillers and pigments used to produce paper. In general, Scots pine is basically only used for chemical pulping. The chemical treatment consists of separating the fibres each other by the cooking process in the digester. (Smook, 1990).

Table 2.9. Raw materials used in paper production, Sweden, 2008; figures are presented in percentage (Skogsindustrierna, 2008)

Raw material	Percentage
Chemical pulp	44
Mechanical pulp	27
Semi-chemical pulp	3
Recovered paper pulp	14
Fillers and pigments	12

Variables to control chemical processes can be distinguish in wood-chips requirements and variables for the cooking process in the digester. Before the cooking process, the variables most important to control are: size, shape, species and mc. The digester is the deposit where wood chips get separated into single fibres. In the digester, the cooking process dissolves lignin and some carbohydrates. Variables of the cooking process consist of type and concentration of the chemical employed, pressure, temperature and cooking time. What influence the most into chemicals reaction with wood are the chemicals and the temperature. Temperature increases or decreases the speed of the reaction. The amount of chemicals added is dependent on the mc in the wood chips. An excess of chemical concentration will destroy the fibres, and a too low chemical concentration will produce uncooked wood chips that need to be removed from the process. In these two cases pulp production will decrease and a loss of pulp will occur. All factors influence to certain extent and must be controlled as accurate as possible. (Haygreen & Bowyer, 1996; Löfstedt, 1996; Smook, 1990).

The mc in wood chips is of importance since it is the dry weight of the fibres which is correlated to the amount of pulp that comes out of the process. A miss-determination of mc can result in erroneous payments of truck loads of the wood chips, in low efficient processes (even losing material), and in increasing energy consumption.

Importance of mc for energy production (wood fuel)

The origin of the wood coming for energy production is most variety. Wood-chips supply comes from different species, different stands, different sizes, different mc, and all this mixed in many cases. The origin can be felling waste, forest industry waste, fuel plantations (mainly *Salix sp.*) and rejected wood from packing. This means that wood-chips mixture contains roots, wood, bark, needles, stones, gravel and GROT (by-product from clear-cutting). Thus mc might vary from 10% to 65%, but mostly mc ranges 45% to 55% (Nyström, 2004). This fact causes difficulties because the desired objective is to have a uniform mc in the incoming flow of wood chips to the process. A high mc in the woodchips means that extra energy is needed for vaporizing the water to steam. A flow of wood chips not uniform in mc can generate problems. When mc suddenly increases significantly, the temperature in the boiler may drop drastically leading to an incomplete combustion which produces monoxide of carbon. (Nyström, 2004; Löfstedt, 1996).

Importance of mc for board production

The raw material received in a board mill can be only roundwood from some wood species, or as complex as roundwood, shavings, chips, mixed mill residues and sawdust. Roundwood supply means usually green material and few species; if the board mill only receives this kind of material, mc homogeneity in the flow of wood chips to the process will be easily achieved. When the supply to the board mill is a variety of materials with a wide range of mc, maximum control is needed in order to minimize adjustments to the process. When entering the mill, raw material must be as uniform as possible in form of species and mc. Or if this is not possible it is necessary at least to maintain a uniform mix of the materials. (Haygreen & Bowyer, 1996).

A proper mc before pressing the particles for board formation is highly necessary. A minimum mc is required in order to give elasticity to the particles, so pressing achieves the wanted result. Higher mc in particles than needed means strong evaporation of water while pressing and produces damages or imperfections to the board. This is why before pressing the mc of particles must be between 2% to 6%. Mc generally must never be above 10% for a proper hot setting resin. (García, *et al.*, 2002; Maloney, 1989; Vignote and Peris, 1996; Ginzel and Peraza, 1966).

2.6 Technologies for measuring mc

The on-going research for a suitable method to determine mc in wood chips is abundant nowadays. Many technologies are available although it is still needed a development of them. Examples of measurement methods are: NIR, Radio frequent, Microwave and Nuclear magnetic resonance and Dual energy x-ray absorptiometry.

Near infrared spectroscopy

NIR stands for Near Infra Red spectroscopy. This method consists of a lamp that illuminates a material with light. Light wave-lengths used are from 850 to 2500 nm. Some of the light is absorbed by the material and some is reflected. There are four absorption maximums of these waves in the mentioned range that are associated with water. A lens collects the reflected wavelengths, and a detector gathers the data producing the NIR spectra. (Nyström & Dahlquist, 2004).

Multivariate calibration is used to analyse the spectra in NIR and to perform calibrations. It is a statistical method where different parameters affecting the measurement are taken into account. It is useful when several factors contribute to the overall observed response. Moreover, it provides with a large number of applications in NIR spectroscopy. In NIR, the spectra can be deformed by variations in density of the material, different chemical composition of the material, mc of the material, variation of distances to the samples, etc. NIR technology only penetrates the material a few centimetres. (Bro, 2003; Nyström & Dahlquist, 2004).

Radio frequent measurements

The system consists of two antennas on both sides of the sample and a network analyser. Electromagnetic radiation produced by the network analyser is emitted by the antenna, reflected by the sample and the signal obtained gets registered and the information is gathered in a spectra. When the electronic radiation reaches the sample due to the polar dielectric properties of bio-fuels, an electric field is created in the sample and reflects a signal back. Temperature, grain direction and cellulose content have a certain degree of influence in dielectric properties. (Nyström & Dahlquist, 2004).

Radio frequency has similar problems than NIR, it gets influenced by variation in density, temperature, etc. Therefore, calibration against samples is performed with multivariate analysis. (Nyström & Dahlquist, 2004).

Microwave

Theory and system of this technology are very similar to radio frequent. A network analyser produces the electromagnetic radiation. The impulse is transmitted by an antenna, goes through the sample and is received by another antenna. Disturbances may appear and can be compensated by nuclear magnetic resonance or by attenuation and other adjustments. Microwave technology suits for flow measurements and to use it off-line. Flow measurements only fits to a practical sample depth of few decimetres. (Nyström & Dahlquist, 2004).

Nuclear magnetic resonance

It could be possible to apply this technology to measure mc in wood-chips. A commercial instrument measuring mc in timber proved feasibility of the method to measure mc in wood, although the range of mc used was 7-18%. This method consists of sensing the amount of hydrogen in a sample using the strength of a magnetic field. (Nyström & Dahlquist, 2004).

Dual energy x-ray absorptiometry

Mantex technology is an alternative for fast determination of mc. It is based on Dual X-ray Absorptiometry technique, which is a technique developed for medical purposes. Its medical application is to determinate bone-mineral and soft-tissue composition of human bodies (Mazess, *et al.*, 1990). It started as a mean to measure bone mineral density, and it is still in an early stage of development; research is ongoing for its improvement and further applications (e.g. for measuring fat content). Proves not to be dangerous, since nowadays it is used for total-body or regional (partial-body) measurements (Mazess, *et al.*, 1990). Quoting to Gilsanz: “DXA is, by far, the most widely used technique for bone measurements. It is low in cost, accessible, easy to use, and provides an accurate and precise quantification of bone mass in adults.” (Gilsanz, 1998).

Mantex technology uses this technique for measuring the mc in a biological material. It is in its early stage of development, and the first implementation was done for measuring mc in

wood. This technology allows determination of mc in samples of wood chips or solid wood. It uses radiation with two different wavelengths of different energy levels, to analyse the item. Figure 2.3.

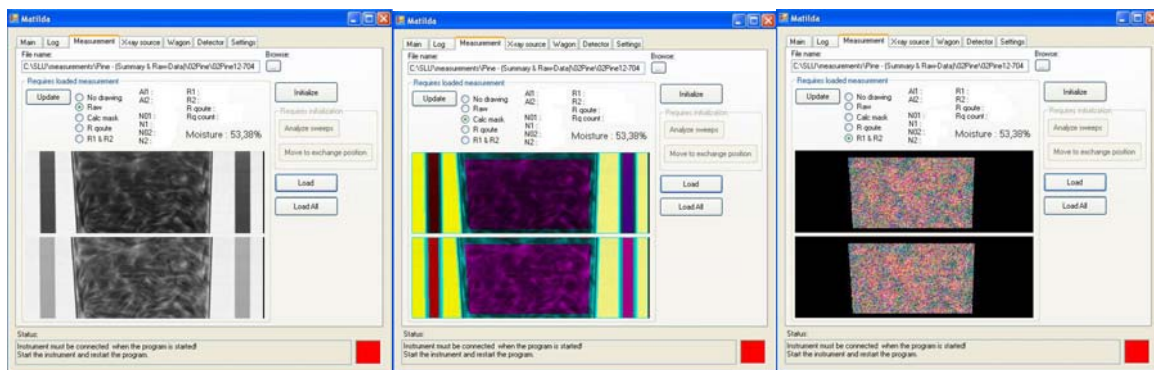


Figure 2.3. Three snapshots from the three different images obtained with the software of the prototype.

By this method, two x-ray beams are produced and are partly absorbed by the material that is to be analysed. The quotient between the energies provides information about the composition of the material. The materials that mostly absorb the beam are atoms of hydrogen and oxygen. The wavelengths have the energies of 40 keVp and 90 keVp. In order to assure stable measurements, two aluminium bars (reference objects) are placed separated at the sides of the sample. When the x-ray tube irradiates the reference objects, the information provided is used to correct variations in the equipment due to wear and temperature changes. The measurements of the reference object consist of an average of ten repeated measurements done in the same position. Measurement of the reference objects occur each time the reference object is at the measuring position, and for both sets of energy. The measurement with the high energy uses a copper filter. Figure 2.4. (Engström, 2007; Hultnäs *et al.*, 2009; Nordell & Vikterlöf, 2000).

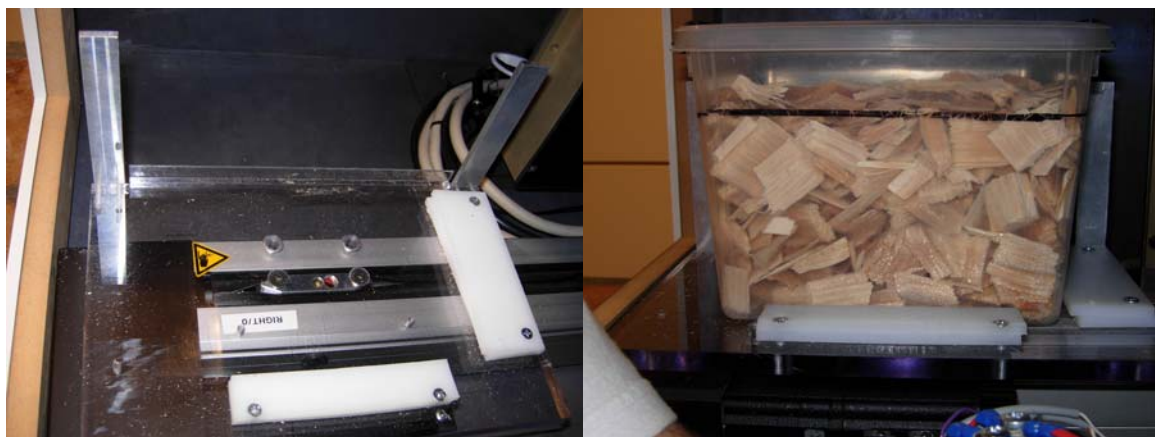


Figure 2.4. Reference objects and sample positioned and ready to be measured.

During the measurement the sample is scanned twice, one scan for each energy level. First, the sample is scanned with a set of lower energy, afterwards, it is scanned with the higher energy using a copper filter to obtain a harder spectrum and bring down the intensity. A wagon moves a platform with the sample and both reference objects through the space in between the x-ray tube emitter and the receiver plate. The reference objects and the sample are placed in the wagon in a way that the reference objects are scanned before and after the sample. When the

wagon starts to move, it stops some seconds to measure the first reference object, moves again scanning the sample, and stops one more time to measure the second reference object. The same procedure is repeated with the other set of energy. (Hultnäs *et al.*, 2009; Nordell & Vikterlöf, 2000).

2.7 Previous study

The study conducted by Engström proved the feasibility of dual energy x-ray technique for measuring mc in wood chips; a common prototype for medical purposes was used in that study (Engström, 2007). Therefore, Mantex developed a prototype that needed to be calibrated against samples and then tested. The department of Forest Products at the SLU in collaboration with Mantex, conducted a study to test and evaluate this prototype. (Hultnäs *et al.*, 2009).

The study consisted of two tests with samples with known mc. Samples used for this research were spruce wood chips which are used for pulp production. The first test was performed in order to calibrate the prototype, and the second test was carried out to evaluate the development of the prototype's software. The reference method used to calibrate and evaluate these tests was the gravimetric method. (Hultnäs *et al.*, 2009).

The first test consisted of a set of 30 samples divided in three series with different mc levels, ranging from 8% to 57% of mc (obtained by the gravimetric method). The samples were dried for different periods of time in order to get the range of mc. After statistical analysis, the results provided the necessary information to calibrate the algorithms of the prototype's software. This calibration was performed by Mantex. In order to evaluate the calibrated prototype the second test was performed with a set of samples divided in nine series of mc between 21% and 57% (obtained by the gravimetric method). Different treatments were done for each series: drying different periods of time (3, 4 and 5 days), maintained in water different periods of time (3 and 5 days), or simply raw wood chips. Every sample was measured 10 times with the prototype, obtaining 300 measurements for the first test and 450 measurements for the second test. (Hultnäs *et al.*, 2009).

The results from the second test, the evaluation of the method, gave a positive result. The statistical analysis rendered the results from a regression line. The Standard Error of Estimate (SEE) was 1.83%, and the Coefficient of Determination (R^2) was 98%, demonstrating that there was a good fit of the data to the model. The average of the Standard Deviation (SD) of the measurements per sample done with the prototype was 0.53%. Correlation of both methods is shown in the Figure 1B. The results from the measurements done with the prototype are shown on the y-axis and the results from the drying method are shown on the x-axis (Figure 2.5). The regression line was $y = 1.14x - 0.05$. The confidence intervals shown in Figure 2.5 were at the level of 95%. (Hultnäs *et al.*, 2009).

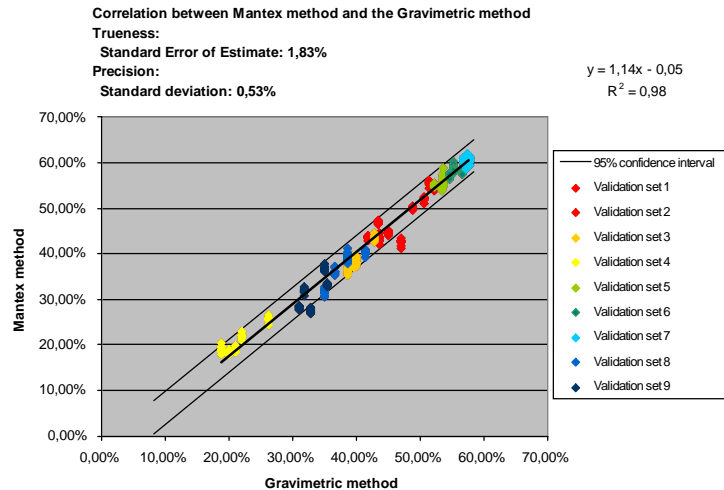


Figure 2.5. Some statistical results, and the regression line with all the validation sets plotted in the graph (Hultnäs *et al.*, 2009).

This study demonstrated that the new technology proved to achieve satisfactorily the expected demands. The result showed low levels of deviation between the tested technique and the reference method. SD and SEE were satisfactorily low. Although a small deviation between the both methods was found, this discrepancy was thus of finite extent. The conclusion was that dual energy x-ray absorptiometry can be used to determine mc in spruce wood chips rapidly and accurately. (Hultnäs *et al.*, 2009).

3. Methodology

3.1 Material

The wood chips used for this study came from sawmill and they were characteristically used in Swedish pulp industry. Sawmill wood chips have a special characteristic which is that they mainly consist of sapwood. They contained small pieces of bark and other pollutants, also some wood chips included parts of bark.

3.2 Equipment

Prototype

The prototype used in this study was the first item developed with the intention to apply dual x-ray technology to the forest industry, in terms of mc measurements in wood chips. Mantex developed the prototype, but Holmen owns the prototype.

The prototype was built within a wood cage, covered by a lead layer in the inside in order to protect against the x-ray. The opening and lid systems were also proved against x-ray and protected against opening action when x-ray where working; the prototype turned off automatically. Size and lead layer turned the prototype quite heavy.

The prototype gave the percentage of mc of the sample directly after a measurement. Not qualified skills were needed to use it; the sample needed to be placed inside correctly, the lid closed and by pressing the GO button, the measurement started. A measurement was very fast, it took about one minute. For repeated measurements a pause was needed between each measurement because the prototype got too warm. The x-ray tube needed to cool down in order to continue measuring; the time required was about 5 minutes.

Oven

The oven used for the study was a Memmert, which is property of the Forest Products Department, SLU. The drying process was performed until the samples reach stable weight.

Scale

The weighing scale used was a Mettler SM 3000, property of the Forest Products Department, SLU. The precision given by the scale was 0.1g of weight.

3.3 Procedure

Collection of samples from the sawmill

Wood chips were collected from Nyby sawmill in Björklinge. They were taken from different places in the pile of wood chips. Initially only two big sacks were collected, but when it was noticed that wood chips were not sufficient, a second collection of samples was done. Five more big sacks were collected to improve the validity of the statistical analysis of the data.

Storage of the samples

The sacks of wood chips were stored in a freezer container, property of the Forest Products Department, SLU. The temperature in the freezer is approximately -26°C , in order to keep wood characteristics stable.

Research design

The study consisted of 7 series, and each of the series was dried different time. The total number of samples was 70, 10 samples per series. The objective was to achieve a wide range of mc levels, and to meet the sufficient number of samples in order to provide the study with valid results. Table 3.1 shows the drying treatment applied to each series. The conditions of the series varied from raw wood chips (dried 0 days) to wood chips dried 5 days.

Table 3.1. Drying time for each series and number of samples per series

Series	Number of samples	Dried (days)
1	10	0
2	10	0.5
3	10	1
4	10	2
5	10	3
6	10	4
7	10	5

The drying treatment consisted of spreading the wood chips on flat surfaces in rooms indoors. The layer of wood chips produced was approximately 2.5 cm thick. Once a day, wood chips were mixed so that they would dry evenly.

Sample description

Wood chips were placed in a plastic container (“Tupper-ware” recipient). The recipient used was the one chosen for Mantex to fit to the prepared place of the sample in the prototype. The volume of wood chips put in the recipient was three liters. A line is drawn in the recipient to mark the minimum volume of wood chips necessary to accomplish a valid measurement of the sample by the prototype. This measure defined the sample size and this fact made sample weight variable. Wood chips could be rather compact or loose in the recipient, then, although they filled the same volume, the amount of wood varies slightly. This fact does not influence the results. The recipient was closed with its cover in order to avoid losing moisture. Figure 3.1.



Figure 3.1. Pine wood chips spread and drying. Sample of pine wood chips prepared.

Testing procedure

Samples have been measured with the prototype 10 times for each sample. Samples weights were measured by a scale when the measurements were performed and once they had achieved a constant weight in the oven Figure 3.2.

Firstly the sample was weighed with the recipient, and afterward the wood chips were taken out of the recipient, it was weighed empty, providing in this way the “initial” sample weight. Samples were dried in paper boxes in the oven at 105 ± 2 °C until the weight was constant (a minimum of 24 hours). This drying procedure was done by following the standard of the Swedish Wood Measuring Association (VMF). It was done according to SS 18 71 70 (Swedish Standard, 1997).

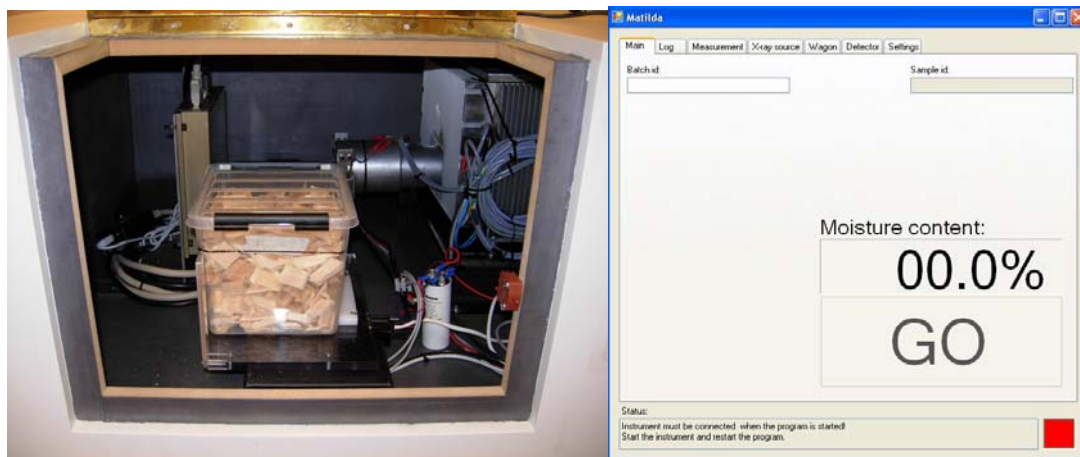


Figure 3.2. Sample positioned in the prototype about to be measured. Snapshot of the software of the prototype, the GO button needed to push to perform a measurement.

Analysis

In this way, 10 mc measurements per sample were obtained with the prototype and one mc measurement was obtained with the gravimetric method. The results achieved with both methods were compared by regression analysis in order to show how well the correlation was between both methods.

The SD calculated is the average of the variances obtained for each ten measurements with each sample. The SD of the total of the series was calculated with the formula (2):

$$\sigma = \sqrt{\frac{\sum((n_i - 1) * \sigma_i^2)}{\sum(n_i - 1)}} \quad (2)$$

n_i = number of measurements per sample done with the prototype.

i = sample number.

σ_i^2 = variance of the measurements per sample.

The mc determined by the gravimetric method was calculated with the formula (1). A correlation of both methods, with a regression line, offered clear results. The programs used for data treatment were Microsoft Excel and Minitab in order to get the statistical results.

4. Results

The 70 samples provide 70 values of mc measured with the gravimetric method. These 70 values are the reference to contrast with the 700 measurements taken with the prototype. The sample with the highest mc has 56.2%, and the sample with the lowest mc has 7.3% (result from the gravimetric measurement). A summary of the measurements is given in Table 4.1 in Appendix 1. In Table 4.2, the samples are gathered in seven series with ten samples in each series and with similar mc. There is a summary of the average of mc measured with the gravimetric method for each series, and with the duration of the drying treatment.

Table 4.2. Treatment of the seven series and the average of mc obtained with the reference method for each series

Series	Dried (days)	Average mc (%)
1	0	55.4
2	0.5	53.2
3	1	41.0
4	2	37.8
5	3	28.1
6	4	18.6
7	5	9.4

The Figure 4.1 gathers all the data obtained in this study in a graphic, providing an overview of all measurements done with both methods. The y-axis gives the mc in percentage, and the x-axis lists the samples in a descending order of mc determined by the reference method. It shows the difference between both methods, and the range width of mc provided in this study.

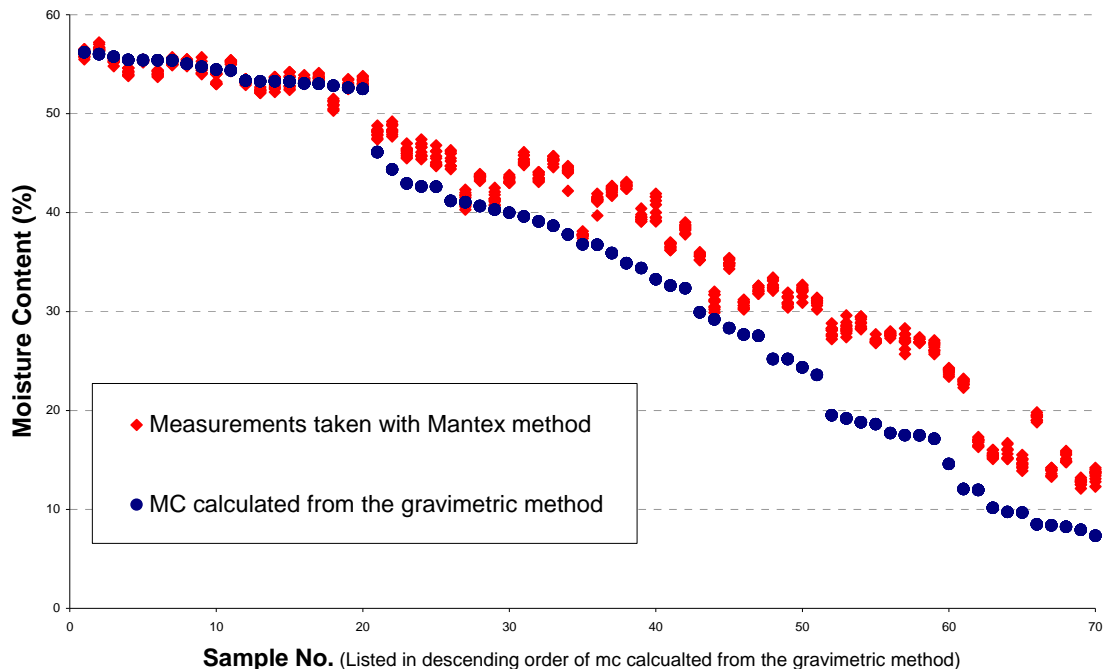


Figure 4.1. Mc values determined by both methods, listed in a descending order of mc measured with the gravimetric method.

The Figure 4.1 shows the measurements of the mc done by the prototype as ten red dots per sample against one blue dot per sample for the ones determined by the gravimetric method. It shows the range of mc that has been covered by this test; from 56.2 to 7.3%. The only two gaps that remain are between the mc levels of 52.5% to 46.1%, and between the mc levels of 23.6% to 19.5%. Moreover, it shows that the prototype measures the mc higher than the reference values. In this graph it is very obvious how close the measurements with both methods are; when the percentage of mc is between 56.2% and 52.5%. For measurements below a mc of 50%, it can be observed how the difference between both methods is increasing. The measurements taken below 12% of mc [samples 60 to 70 listed in descending order of mc measured with the gravimetric method] show that the difference between both methods is smaller again. This can be better appreciated in Figure 4.2.

In the Figure 4.2, the difference in absolute value between the measurements of both methods is at the y-axis. Percentage of mc calculated from the gravimetric method is found at the x-axis. It is possible to notice how the absolute error is changing when the wood-chips measured are getting drier. It shows that the difference between the measurements of both systems is increasing while the percentage of mc is decreasing.

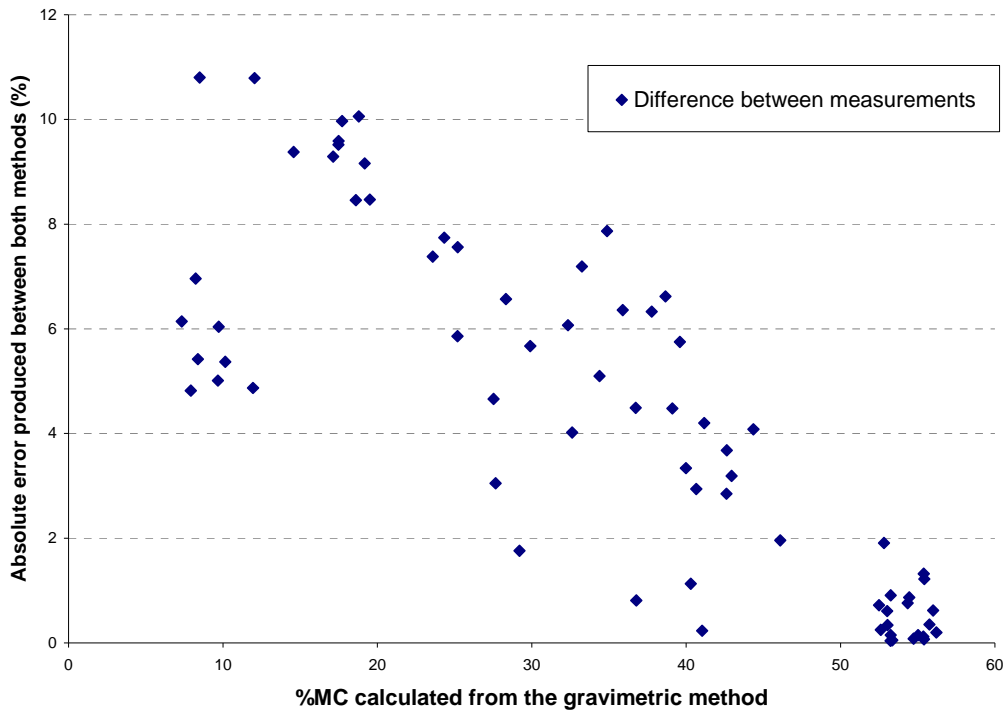


Figure 4.2. Absolute error between both methods, listed in descending order of mc measured by the gravimetric method.

In order to analyze the trueness of the given test, statistical analysis of the data and a regression line have been the selected options. The Figure 4.3 shows the correlation between Mantex method and the gravimetric method. All the measurements are plotted with the regression line that shows how it fits to them. It has been checked if there were some outliers, however, the conclusion is that the data is accurate and has no outliers of importance.

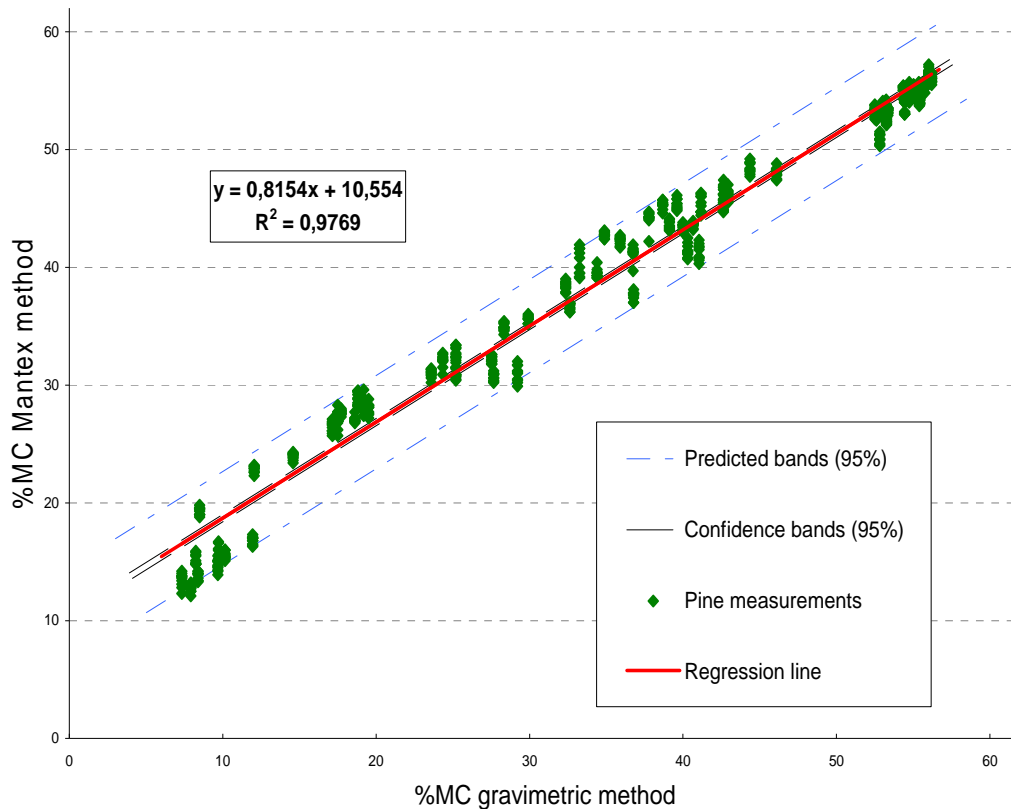


Figure 4.3. Correlation between Mantex method and the gravimetric method (data set, regression line and confidence and predicted bands).

Regression Analysis (Figure 4.3):

The fitted line reflects the trend in the data. The two variables introduced are: the percentage of mc measured with the prototype, and the percentage of mc measured by the gravimetric method.

The following shows the statistical analysis of the discussed data:

Correlation coefficient (R): 0.99

Coefficient of determination (R-squared): 0.98

Standard Error of Estimate: 2.01%

Standard Deviation: 0.45%

The regression equation is:

Mantex method = 10.554 + 0.8154*Gravimetric method

Table 4.3. Statistical analysis of the coefficients from the regression equation

Predictor	Coefficient	SE Coefficient	t-value	P-value
Constant	10.5543	0.1819	58.03	0.000
Drying method	0.815365	0.004749	171.68	0.000

Table 4.3 shows that the coefficients of the regression equation are valid. In comparison to the coefficients, the standard errors of the coefficients are low which shows good precision for both coefficients. The t-values show that coefficients are significant. And the very small p-

values prove a reliability of both coefficients for the regression equation (it proves that correlation is not due to random sampling). The correlation coefficient, R , ranges from -1 to +1. An R of +1 would be the best correlation coefficient, and an R of 0 would mean that there is no linear relation between the two variables. In this study, the R is 0.99 which is very close to 1; it shows that the two variables are strongly related. The coefficient of determination, R^2 , ranges from 0 to 1. The higher the R^2 is, the better the model fits the data. In our case R^2 is 0.98 proving that the regression line approximates well to the real data points. The SD measures the variability or dispersion of the data set. In this study, the SEE is 2.01%, which indicates that the data points tend to be very close to the same value.

The area enclosed in between the confidence bands shows the area in which it is 95% sure that the fitted-line (regression line) is included. This area gives a visual sense of how well the data define the best-fit curve. For the data obtained in this test, the confidence bands are very close to the regression line, which proves a high accuracy of relation between the regression line and the data. The 95% prediction bands enclose the area in which it is expected to enclose 95% of future data points. This includes the uncertainty in the true position of the curve, and also accounts for the data scattered around the curve. Therefore, prediction bands are always wider than confidence bands. In this study, the 95% prediction bands adjust quite good, enclosing a small area. Moreover, almost all the measurements done with the prototype are included in the area limited by the prediction bands.

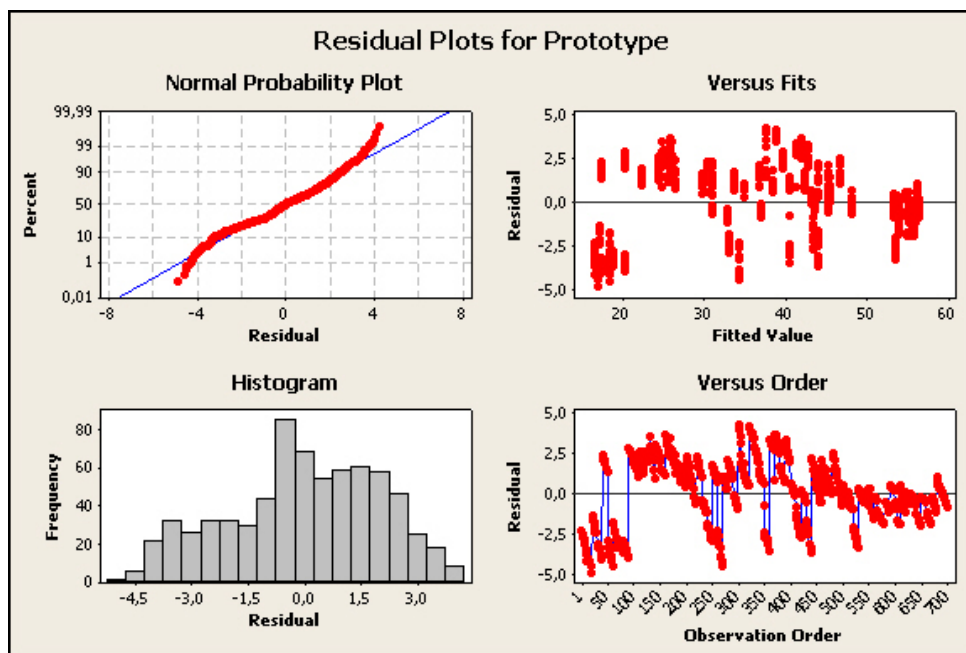


Figure 4.4. (Regression diagnostics) Residual plots for Mantex method.

Figure 4.4 shows the residual plots in order to examine the well suited-values of the model to fit in the regression. It includes a normal probability plot, a histogram, residuals versus fitted values and residuals versus order of data. The normal probability plot shows that the residuals differ from normality in the extremes producing two tails. Despite that the data proves to be near linear.

Table 4.4 lists all the unusual observations for standardized residues larger than 2.0, in absolute value. The standardized residues measure how big the standard error from each observed value of the mc measured with the prototype deviates from the fitted-line. In this

study, there are 18 observations which have standard residues larger than 2.0; however there is no standard residues larger than 3.0. Moreover, the great majority of the standard residues are larger but close to 2.0. The homogeneity of the values of the residuals proves that there are no outliers of importance.

Table 4.4. Unusual observations

Obs.	Gravimetric method (x)	Prototype (y)	Fit (y predicted)	SE Fit (SE of y predicted)	Residual	St. Residual
10	7.3	12.3	16.5310	0.1510	-4.2310	-2.11
13	7.9	12.9	17.0120	0.1485	-4.1120	-2.05
14	7.9	12.9	17.0120	0.1485	-4.1120	-2.05
15	7.9	12.8	17.0120	0.1485	-4.2120	-2.10
16	7.9	12.8	17.0120	0.1485	-4.2120	-2.10
17	7.9	12.7	17.0120	0.1485	-4.3120	-2.15
18	7.9	12.5	17.0120	0.1485	-4.5120	-2.25
19	7.9	12.5	17.0120	0.1485	-4.5120	-2.25
20	7.9	12.1	17.0120	0.1485	-4.9120	-2.45
40	8.4	13.3	17.3871	0.1467	-4.0871	-2.03
58	9.7	14.3	18.4471	0.1414	-4.1471	-2.06
59	9.7	14.2	18.4471	0.1414	-4.2471	-2.11
60	9.7	13.9	18.4471	0.1414	-4.5471	-2.26
269	29.2	30.2	34.3711	0.0806	-4.1711	-2.07
270	29.2	29.9	34.3711	0.0806	-4.4711	-2.22
301	33.3	41.9	37.6652	0.0765	4.2348	2.10
321	34.9	43.1	38.9943	0.0761	4.1057	2.04

5. Discussion

This study was performed in order to verify how the prototype measures mc in pine wood chips. There is no great difference between spruce regression line after prototype calibration and pine regression line without any further calibration.

In order to show how results approach to the reference method, regression lines are represented in Figure 5.1. The graph shows: the regression line of the results of this study (pine's regression line), a perfect correlation between both methods (perfect correlation line) and the regression line of the previous study performed with spruce (spruce's regression line). This allows to compare the pine study and the spruce study performed by Hultnäs *et al.*, (2009) with an ideal measuring system. The equation of the perfect correlation line is $y = x$; each measurement made with the prototype would give the exact value that the gravimetric method gives. Similarity to the perfect correlation is achieved in pine for mc from 52% to 56%.

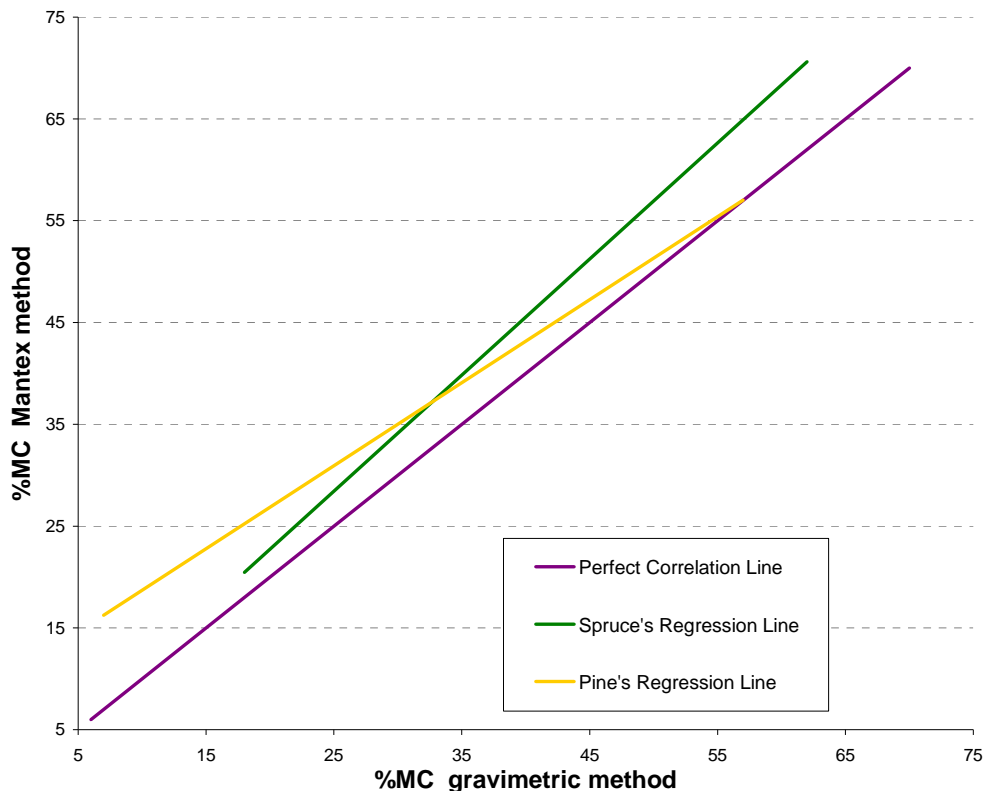


Figure 5.1. Correlation of Mantex method and gravimetric method; regression lines to compare them.

The equations of the regression lines are:

Pine's: $y = 0.815 * x + 10.55$

Spruce's: $y = 1.140 * x - 0.05$

The differences in step when they are compared to the perfect correlation line are 0.185 for pine's line and 0.14 for spruce's line. Both of them are relatively close to the correct value. The y-intercept of spruce's regression line (-0.05) is very close the perfect correlation line value. On the other hand, y-intercept of pine's regression line (10.55) is relatively far from the perfect correlation line value.

Both regression lines have different mc ranges 21% to 57% for spruce and 7% to 56% for pine. Both regression lines stay over the perfect correlation line indicating that the prototype overestimates the values of mc compared to the reference method. Although, in pine's regression line, the data for high mc (52.5%-56.2%) shows close values to a perfect correlation. This fact indicates that the prototype could achieve same values of mc as the gravimetric method; with of course certain deviation/dispersion added.

Measurements of mc made with the prototype show a low dispersion with low SD of 0.45%. Regression analysis shows satisfactory results; a relatively low SEE (2.01%), and the R² value (98%) demonstrates that the model fits to the data. Statistical results from the study performed with spruce are very similar to the results of this study, Table 5.1. The prototype was not calibrated against pine before this study was conducted so better results can be expected after an adequate calibration of the prototype, since SD and SEE are very low. The algorithm can be developed from the data obtained in this study and from the absorption fractions stored in text files of the prototype. Once the developed algorithm is incorporated into the software of the prototype, results of measuring pine will probably achieve better accuracy.

Table 5.1. Comparison of the statistical results obtained from both studies. (Hultmäk et al., 2009)

Descriptor	Pine's results (%)	Spruce's results (%)
R ²	98	98
SEE	2.01	1.83
SD	0.45	0.53

Composition of pine is different to spruce composition and that may explain the different regression line obtained. The different contents in cellulose and lignin of both species could influence; Scots pine has less cellulose content, but more lignin and other polysaccharides contents than spruce. The extractives in pine are more than double of the extractives for spruce, and spruce has a higher content of residuals. Bark content might also influence in some extent, since bark composition probably varies between both species. Bark contents also water, and it dries faster than wood and may introduce the unexpected errors throughout the range mc.

A certain deviation of the results might be explained by a limitation of the prototype. In contrast to the gravimetric method where the whole sample was measured, the prototype cannot measure the whole sample due to several reasons. The first reason is the shape of the radiation beam; it does not allow all beam to reach the receiver. The second reason is that the recipient of the sample has rounded corners, and if the whole sample were measured, these corners would introduce an error difficult to quantify.

In this study, only wood chips coming from sawmill were used, they mainly consist of sapwood, and an unexpected error might be introduced when measuring sapwood and heartwood together. Calibration of Mantex technology would be then needed.

Apparently Mantex technology could reach the mentioned goals for the forest industries. It assures stable measurements at the moment of measuring, correcting any possible variations in the equipment or temperature changes. Deviation in the correlation between both methods is likely to be easily corrected. Further research on this technology with wood of other species, or for other materials, and in different conditions would be recommended to be studied; and so develop further its possibilities.

6. Conclusions

The evaluation of dual energy x-ray absorptiometry when determining mc in pine wood chips provides the following conclusions:

- Measurements of mc made with the prototype show a low dispersion with low standard deviation of 0.45%.

- Regression analysis shows satisfactory results; a relatively low standard error of estimate (2.01%), and the R^2 value (98%) demonstrates that the model fits to the data.

- Different regression lines between spruce and pine shows the necessity to develop a different algorithm for measuring mc in pine wood chips.

- Since standard deviation and standard error of estimation are very low, it is likely that after a calibration, measuring mc in pine wood chips would give similar results in terms of deviation as for spruce.

- It is worth pointing out the fast measuring of mc with the prototype; each measurement takes about one minute.

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Appendices

Appendix 1. Table 4.1

The table shows the results of the measurements done for each and every sample, classified by series. For each of the samples, it shows the average of the ten measurements of the mc taken with the prototype, and the mc calculated from the gravimetric method.

Series No.	Sample No.	Percentage of Moisture Content	
		Prototype (average)	Gravimetric Method
1	1	56.63	56.01
	2	56.01	56.21
	3	55.34	55.41
	4	55.25	55.37
	5	55.41	55.76
	6	54.21	55.43
	7	54.66	54.74
	8	54.08	55.40
	9	55.12	54.36
	10	55.18	55.03
2	11	53.39	53.05
	12	52.86	52.61
	13	53.22	52.50
	14	53.30	53.35
	15	53.28	53.24
	16	53.63	53.02
	17	50.91	52.82
	18	52.35	53.26
	19	53.58	54.45
	20	53.11	53.26
3	21	43.60	40.66
	22	48.06	46.10
	23	45.47	42.62
	24	48.44	44.36
	25	46.32	42.64
	26	45.35	39.60
	27	44.11	37.78
	28	42.75	34.88
	29	45.29	38.67
	30	46.13	42.94
4	31	41.43	40.30
	32	41.27	41.04
	33	37.59	36.78
	34	43.33	39.99
	35	43.59	39.11
	36	36.64	32.62
	37	45.37	41.17
	38	41.23	36.74
	39	39.49	34.39
	40	42.26	35.90

Series No.	Sample No.	Percentage of Moisture Content	
		Prototype (average)	Gravimetric Method
5	41	32.19	27.53
	42	30.97	29.21
	43	34.90	28.33
	44	30.71	27.66
	45	32.08	24.34
	46	30.97	23.59
	47	32.76	25.20
	48	35.58	29.91
	49	40.44	33.25
	50	38.42	32.35
6	51	27.09	17.50
	52	27.00	17.48
	53	28.86	18.80
	54	28.34	19.18
	55	27.98	19.51
	56	27.68	17.71
	57	26.43	17.14
	58	23.96	14.58
	59	27.07	18.61
	60	31.05	25.19
7	61	15.52	10.15
	62	16.82	11.95
	63	22.84	12.05
	64	15.76	9.72
	65	14.69	9.68
	66	13.80	8.38
	67	12.74	7.92
	68	13.47	7.33
	69	15.20	8.24
	70	19.29	8.49

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