

# Dual-Energy X-Ray Absorptiometry Analysis for the Determination of Moisture Content in Biomass

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The aim of the present study was to describe and validate a novel method able to fast, accurately and with a high precision determine the moisture content of biological materials ranging 10% to 70%. A calibrated Dual X-ray photon absorptiometry technique is validated against moisture content in wood chips. Conventional criteria such as the Pearson correlation coefficient, the root mean square errors of calibration and coefficient of variation were used for the evaluation of the method. The method is independent of the thickness and density of the object but dependent of the effective atomic number of the material. The results shows that this photon absorptiometric method gives accurate (<2%) and precise (<1%) results for moisture content determination in wood chips.

**Keywords:** Moisture Content, Photon Absorptiometry, Gravimetric Method, Dual Energy X-Ray Absorptiometry, Spruce Chips, Pine Chips.

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## 1. INTRODUCTION

Previously known methods to estimate the amount of moisture content in wood material with reasonable good accuracy (<1–2%) involve drying of the material.<sup>1</sup> These gravimetric methods using hot air are often used as the standard method for moisture content determination.<sup>2,3</sup> However, such methods are cumbersome and tedious, and it would normally take a day or more until a correct measured value could be obtained, which delays the overall processing. It is therefore a need for a fast and reliable method to estimate the moisture content.

Similar needs exist in other industries handling biological material. For example, it would be advantageous to have a fast and reliable method for estimating the moisture content of biomass in the bio energy field, in order to control the burning process more precisely, and improve its efficiency.

Water can be found in most biological and non-biological material in different forms. Due to their hygroscopic nature, biological materials can also gain or lose moisture depending on the material-air interaction that take place during storage. In addition to the gravimetric method to measure the moisture content there is near infrared and microwave technology.<sup>4,5</sup> Also capacitance gauges may be

used as well as other electrical devices, like conductivity measurement.<sup>6</sup> The limitation of above-mentioned moisture measurement technologies is often the dominant influence of surface moisture content to the end result. Also ambient conditions for example snow and ice or temperature below zero, may decisively disturb the determination. The use of photon absorptiometry has the potential to overcome the difficulties with present day methods since the radiation has penetrating power and snow and ice will not affect the moisture measurement.

Photon absorptiometry methods have been used in medical applications, mainly for the determination of bone mineral content in the skeleton. Single X-ray absorptiometry has been used for measurements on the peripheral skeleton and dual X-ray absorptiometry for measurements on the axial skeleton.<sup>7</sup> Photon absorptiometry is sensitive to the effective atomic number and density of the material being analyzed.<sup>8</sup> The absorption and scattering of photons in the energy range that is most convenient for material analysis is a combination of two photon-matter interactions. These two are called the photoelectric effect and Compton scattering and are energy dependent.

In this work, a new method, using two different photon X-ray energies from an X-ray tube will be presented. With this method it is possible to determine the moisture content in most biological materials for instance crops, cotton, pulp and wood. In this paper the application of the method for moisture content in wood chips is described.

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## 2. MATERIALS AND METHOD

A Mantex desktop scanner (Mantex AB, Kista, Sweden) has been used for the absorptiometric method measurements in this study. This device consists of an X-ray tube operated at 90 kVp, 2 mA and 40 kVp, 5 mA. This gives that the effective energies was about 58 and 30 keV. The detector consisted of a semiconductor linear array detector.

The samples were contained in plastic boxes about 15 cm in width and 21 cm in length and 17 cm in height. The volume was about 3 liters. These boxes were scanned once for each energy with a total measurement time of 60 seconds. The measurement setup is shown in Figure 1.

The method has been calibrated for measurements of spruce, pine, spruce and pine mixture and pine bark. The spruce and pine mixture consisted of about 50% spruce wood chips and 50% pine wood chips. Samples of about 0.5 m<sup>3</sup> were collected and divided into subsamples of about 3 liters. The different moisture levels were achieved by air drying. The subsamples were for this purpose spread on metal plates and allowed for drying several hours before measurement. Before measuring the subsamples they were homogenized in a closed bucket in order to achieve a homogenous sample.

Figure 2 shows the measurement equipment used for this study.

The Desktop scanner used in this study was lead shielded in such a way that the dose equivalent rate does not exceed 0.2 μSv/h at a distance of 10 centimetres at any point around the box. This could be compared to the dose rate from natural background radiation in a normal environment in Sweden which is about 0.4 μSv/h. A switch is mounted at the lid of the measurement space. This turns off the X-ray equipment if the lid is opened during operation. The dose rate from the Mantex desktop device and the design of the box shows that the device is an enclosed installation.

## 3. THEORY/CALCULATION

We assume that both photon energies are attenuated exponentially and thus the condition narrow beam geometry is valid.

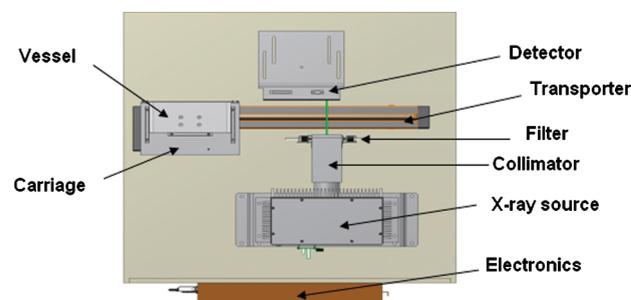


Fig. 1. Schematic drawing of the measurement setup.



Fig. 2. A Mantex desktop scanner was used for the measurements in this study.

The following relations are then valid:

$$N_1 = N_{0,1} \exp(-\mu_1 X) \quad (1)$$

$$N_2 = N_{0,2} \exp(-\mu_2 X) \quad (2)$$

Where  $N_i$  is the observed transmitted countrate for energy  $i$ ,  $N_{0,1}$  is the countrate for energy  $I$  with no attenuator,  $\mu_i$  is the linear attenuation coefficients for energy  $I$  and  $X$  is the thickness of the attenuator.

Equations (1) and (2) can also be written:

$$\ln(N_{0,1}/N_1) = \mu_1 X \quad (3)$$

$$\ln(N_{0,2}/N_2) = \mu_2 X \quad (4)$$

The countrates without attenuator could be determined besides the object or attenuator.

From Eqs. (3) and (4) an expression called  $k$  can be derived

$$k = R_1/R_2 \quad (5)$$

where:  $R_1 = \ln(N_{0,1}/N_1)$  and  $R_2 = \ln(N_{0,2}/N_2)$  and therefore

$$k = \mu_1/\mu_2 \quad (6)$$

The expression (6) is independent of the thickness and density of the object. It is only dependent of the linear attenuation coefficient. The linear attenuation coefficient is dependent upon the effective atomic number of the target material and the energy of the radiation.<sup>8</sup> This comes from the fact that the attenuation of different materials depends on the atomic number of the constituents and not the chemical composition of these constituents. In the energy region used in this paper the photoelectric absorption is important and the absorption per electron depends on about the third power of  $Z$ , where  $Z$  is the atomic number.

The effective atomic number of a compound can be defined from the following expression:<sup>9</sup>

$$Z_{\text{eff}} = {}^{2,94} \sqrt{(f_1 \times (Z_1)^{2,94} + f_2 \times (Z_2)^{2,94} + f_3 \times (Z_3)^{2,94})}$$

where  $f_1$ ,  $f_2$  and  $f_3$  are the fractional contents of electrons belonging to elements  $Z_1$ ,  $Z_2$  and  $Z_3$  respectively.

Since the effective atomic number of a mixture of different elements will be dependent on the amount of water it is possible to use the expression (5) to determine the amount of moisture in different materials where the only variable component is the water content. When the moisture content varies the effective atomic number will vary and thus the linear attenuation coefficient will vary.

By plotting the change in  $k$  as a function of moisture content linear regression could be used to fit the model.

Calibration measurements have been carried out on samples of different biomasses. For these calibration measurements the correct moisture contents have been determined by the gravimetric method.<sup>2</sup> For each material 4 or 5 different samples have been measured at least 3 times and dried in several steps. Final drying of all samples have been in an oven at  $105 \pm 2$  degrees Celsius for at least 24 hours and weighed. The samples are completely dried when the weight is constant within 1 hour.

Moisture content has been calculated according to the formula:

$$MC\% = ((\text{Wet weight} - \text{Dry weight})/\text{Wet weight}) * 100$$

The reproducibility was measured using the coefficient of variation ( $CV = \text{std}/\text{mean} * 100\%$ ) for 20 measurements on a sample of spruce chips after allowing the device to warm up.

In order to evaluate if the measurements are affected if the samples are frozen measurements were carried out on both frozen and unfrozen samples.

## 4. RESULTS

The parameter  $k$  has been determined for 4 different samples of spruce wood chips with different amounts of water, see Table I. The 4 samples have been dried in five steps and measured three times at every step. The moisture

contents were determined by weighing of the samples at each drying step and also when the samples had been completely dried. From these measurements a calibration could be made by plotting the  $k$ -values against water or moisture content, see Figure 3.

The reproducibility measurements on 20 spruce chips samples gave a precision of 0.9%.

In Table II are a few different materials and the linear relationship between the moisture content and  $k$ -value presented.

In order to validate the regression analysis measurements were done on separate samples consisting of a spruce (70%) and pine (30%) mixture. The results are plotted in Figure 4.

The materials in Table III were measured frozen and then again after defrosting. No significant differences were found between frozen and not frozen samples at a confidence level of 95%. The mean difference was 0.26% with a standard error of estimate (SEE) of 0.7%.

## 5. DISCUSSION

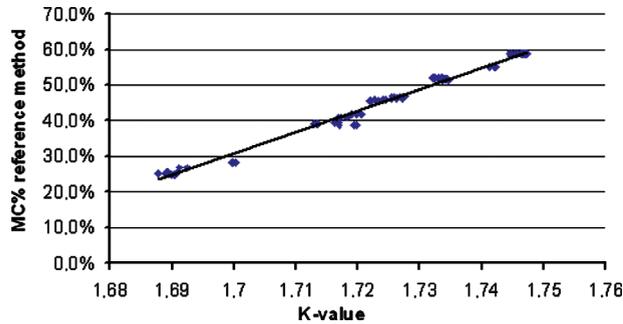
This report describes a novel method for moisture content determination in biological samples.

The results show that a linear calibration equation, albeit with slightly differing coefficients for different materials, provides excellent fit ( $r^2$  values over 0.85). The method advantage is usability under many circumstances. The results are for example not affected if the materials are frozen, it is a fast method and measures through the material. The transmission measurement covers the entire material cross-section in the path of the photons and therefore representative results will be achieved even when the moisture is not homogeneously distributed. This measurement technology is non destructive and also contactless and therefore does not influence the material or wear of the measuring system.

The results for moisture measurements of pine bark were somewhat worse compared to spruce and pine. This

**Table I.** Moisture measurements on four samples of spruce wood chips.

Sample 1 (%)	$k$	Sample 2 (%)	$k$	Sample 3 (%)	$k$	Sample 4 (%)	$k$
58.67	1.74687	58.75	1.74657	58.61	1.74665	58.72	1.74625
58.67	1.74697	58.75	1.74666	58.61	1.74539	58.72	1.7467
58.67	1.74721	58.75	1.7465	58.61	1.74707	58.72	1.74658
51.81	1.7331	51.77	1.73436	51.63	1.73409	51.38	1.73466
51.81	1.7321	51.77	1.73396	51.63	1.73423	51.38	1.7336
51.81	1.73373	51.77	1.7344	51.63	1.73424	51.38	1.73304
45.76	1.72402	46.04	1.72552	46.70	1.72718	45.53	1.72198
45.76	1.72267	46.04	1.72707	46.70	1.72583	45.53	1.72323
45.76	1.72428	46.04	1.72618	46.70	1.72755	45.53	1.72217
38.55	1.71981	38.93	1.71283	41.88	1.71903	39.39	1.71676
38.55	1.71686	38.93	1.71366	41.88	1.71965	39.39	1.71618
38.55	1.7194	38.93	1.71316	41.88	1.72042	39.39	1.71701
24.92	1.68982	25.31	1.68776	28.53	1.69962	25.33	1.68909
24.92	1.69039	25.31	1.68887	28.53	1.70021	25.33	1.68931
24.92	1.69027	25.31	1.68776	28.53	1.69993	25.33	1.69095



**Fig. 3.** Moisture content in spruce wood chips measured by the gravimetric method and plotted as a function of  $k$ -values. The correlation coefficient  $R^2$  became 0.99 and the regression equation:  $MC\% = 6.01 * k - 9.906$ .

**Table II.** Linear relationship between moisture content (MC%) and  $k$ -values, correlation coefficients and standard error of estimate (SEE) for spruce, pine, pine bark and mixture of spruce and pine chips.

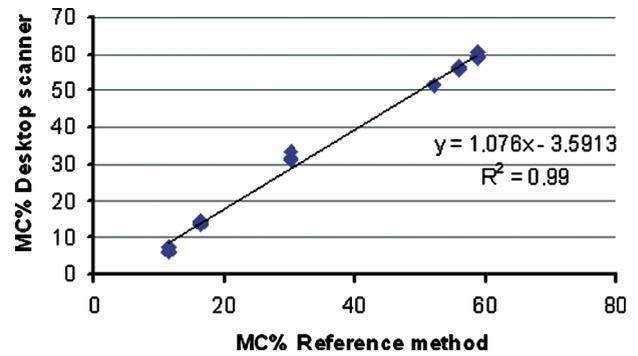
Material	Linear equation	$R^2$	SEE (%)
Spruce	$MC\% = 6.01 * k + 9.906$	0.99	1.2
Pine	$MC\% = 6.29 * k + 10.390$	0.98	1.8
Pine bark	$MC\% = 12.5 * k + 21.712$	0.85	3.9
Spruce and pine mixture	$MC\% = 6.10 * k + 10.067$	0.99	1.3

could be an effect of the more inhomogeneous samples from bark. The bark samples contained also soil and small amounts of gravel.

The precision shows that the method has a good reproducibility and is well suited for repeated measurements of the same object.

Since this method is independent of the density and thickness of the material it could also be adapted to measurements on belt drives and in tubes. Further studies are needed in order to evaluate these applications. Other applications of this technology may be moisture determination in paper pulp and in different biomasses in agriculture.

The reference method to which the photon absorptiometric method has been compared was the gravimetric method.<sup>2</sup> This method has an accuracy of better than 2% and a precision of better than 1%. Both the precision and accuracy of the Mantex absorptiometric device were similar to these figures. However, the oven drying method is not an absolute method since biomass frequently contains varying amounts of volatile compounds which may evaporate while oven drying.<sup>10</sup> In order to quantify the errors inherent in the oven drying method a study where resinous wood species are compared to non-resinous species/samples may be undertaken. Since the accuracy of the absorptiometric method described in this paper is based on the results of the gravimetric method the errors inherent in the latter are superimposed the absorptiometric method. The accuracy of the absorptiometric method will therefore be better than the present results shows.



**Fig. 4.** Validation of regressions analysis on samples of a mixture of spruce and pine chips. The Moisture content measured by the desktop scanner was compared to the moisture content by the gravimetric method. The correlation coefficient  $R^2$  became 0.99 and the standard error of estimate (SEE) 1.9%.

**Table III.** Comparison between measurements of frozen and not frozen samples.

	Moisture content %		Delta (%)
	Frozen	Not frozen	
Pine	12.9	13.4	-0.5
Pine	12.8	13.5	-0.7
Pine	9.6	8.9	0.7
Pine	10.7	11.2	-0.5
Spruce	48.8	48.8	0.0
Spruce	47.1	47.1	-0.1
Spruce	31.1	31.2	-0.1
Spruce	14.8	14.6	0.2
Pine bark	65.9	67.9	-2.0
Pine bark	66.7	66.3	0.4
Mean	32.0	32.3	0.3

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