J Nondestruct Eval (2013) 32:413–417
DOI 10.1007/s10921-013-0195-7

Quantitative Determination of Density and Mass of Polymeric Materials Using Microfocus Computed Tomography

A. du Plessis · M. Meincken · T. Seifert

Received: 10 June 2013 / Accepted: 1 August 2013 / Published online: 29 August 2013

Abstract A density calibration of homogeneous polymeric materials in the range of 0.9 to 2.2 g/cm^3 with micro Computed Tomography (CT) scanning was devised and its accuracy, repeatability and potential sources of error were investigated. The density of unknown materials could be determined successfully in many cases with this calibration. However, in some cases the experimental values deviated significantly from the actual values. These deviations could be attributed mainly to chemical compositional differences compared to the calibration materials. Dual energy CT could be used to determine whether a material is within the range of atomic composition of the calibration materials, in which case the calibration function is accurate. If a material is outside the chemical composition range determined by dual energy CT, the calibration is not applicable and accurate density cannot be determined.

Keywords Quantitative CT scanning · Polymers · X-rays · Computed tomography · Non-destructive testing · Beam hardening · Dual energy CT

1 Introduction

CT scanning is frequently applied in material science to observe material contrasts due to different densities. It is, however, rarely used to determine absolute density values. There have been selected studies where CT densitometry techniques have been used to determine physical density values with the calibration of standard phantoms [1, 2]. Significant effort has been made to accurately quantify bone mineral density with medical CT scanners, as for example, described by Lang et al. [3]. This is also sometimes called osteodensitometry and is not truly a measure of density but rather a quantification of calcium. Similarly, this has been used to measure the mineral density in teeth [4]. Several studies have focused on wood densitometry, as described in [5–7]. This method was even recently used to estimate the density of wood from ancient violins [8]. A recent study in our group focused on the quantification of wood density changes due to thermal degradation [9].

Though the application of micro-CT to the analysis and three dimensional visualization of polymer and composite materials has been widespread (see for example [10, 11]), this is the first demonstration of the technique for the absolute density determination of such materials.

The main difficulties of CT-densitometry are the accuracy and repeatability. Therefore we used a calibration set of different polymer disks ranging in density from 0.9 to 2.2 g/cm^3 to obtain a calibration function from which the density of unknown objects could be determined.

The experimental results highlight the applicability and the accuracy of the CT-determined density. Furthermore they provide guidelines how best to achieve accurate results. An accurate density estimate can potentially be used to identify unknown materials. The limitations of the method were investigated and are reported below.

2 Experimental Setup

A commercial microfocus X-ray computed tomography system was used: the General Electric Phoenix V|Tome|X

A. du Plessis (✉)
Central Analytical Facilities, Stellenbosch University,
Stellenbosch 7602, South Africa
e-mail: anton2@sun.ac.za

A. du Plessis · M. Meincken · T. Seifert
Department of Forest and Wood Science, Stellenbosch University,
Stellenbosch 7602, South Africa

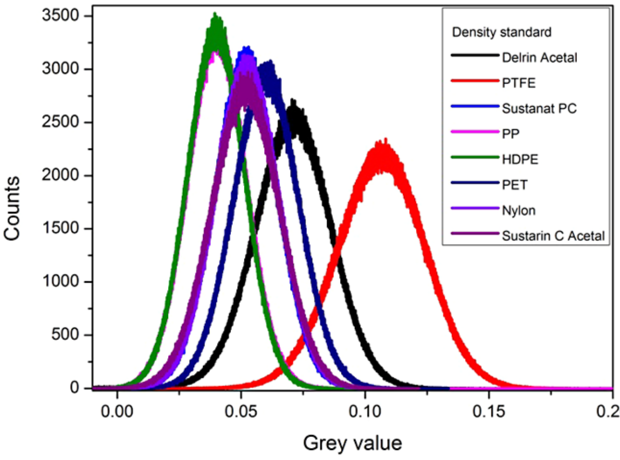

Fig. 1 Histograms of the grey value distribution in the calibration disks

L240. Image acquisition was in the range 500 ms to 1000 ms acquisition time per image, using 500–3000 images per CT scan depending on the magnification. The detector calibration is only valid at a fixed source-detector distance as this distance influences the amount of X-ray radiation reaching the detector and therefore affects the calibration function. In order to compensate for X-ray emission variations and to obtain reproducible values for the calibration function, a series of 1 hour calibration scans were done over a 5 day period.

The polymers used as calibration standards were acquired from Maizey Plastics, South Africa and The Plastic Shop, UK. The used polymers were delrin acetal, polytetrafluoroethylene (PTFE), sustanat polycarbonate (PC), polypropylene (PP), high density polyethylene (HDPE), polyethylene terephthalate (PET), nylon and sustarin C acetal.

Disks of 10 mm thickness and 25 mm diameter were stacked on top of each other and placed on the rotation mount of the system. The reconstructed data was analyzed with the imaging software VGStudio Max 2.1. A representative volume from each disk was selected from the resulting data set to obtain an average grey value for each disk. Each voxel has an associated grey value depending on the material's density and atomic number. The average grey value of the disk is therefore a measure for its density. The histograms for the 8 calibration samples are shown in Fig. 1.

To generate a calibration function, the average grey value of each polymer disk was plotted against the actual density value. The resulting linear function as shown in Fig. 2, resulted in the following regression fit (for 32 bit data):

$$\text{Actual density} = 19 \cdot \text{Grey value} + 0.20$$

This equation can be used to determine the density of an unknown polymeric object from its average grey value, if it is scanned under the same experimental conditions as the calibration function was obtained. It must be noted that two polymers lie above and below the calibration curve, resulting in a better than expected regression fit. This is an inherent limitation in the method and results from potential chemical compositional differences.

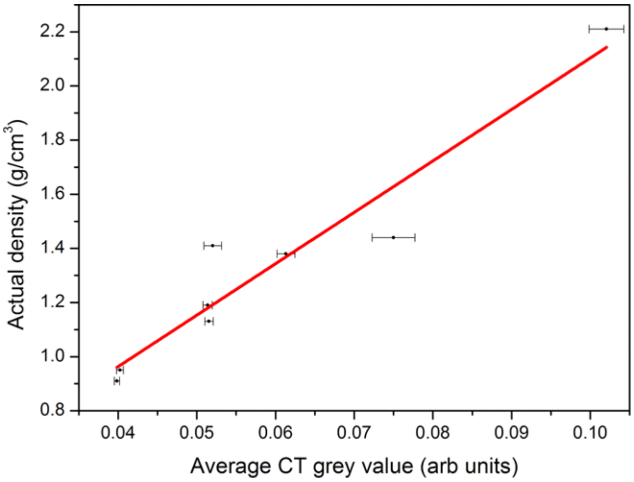

Fig. 2 Linear calibration function for the series of 8 polymers, $R^2 = 0.91$

The values used to fit the measured and actual density are the average of 10 scans obtained over a period of one week (1 hour per scan) and the error bars are their standard deviation. The small error bars show that the values are reproducible and the variation over time is small. These small variations can be explained by slight variations in X-ray intensity due to target damage in the X-ray generation process, electron beam focusing variations, filament aging or current variations, detector temperature or saturation effects, amongst others. It is noted here that this analysis was done with the most accurate reconstruction data using 32 bit grey value data. Decreasing the data type to 16 bit did not change the accuracy but improved the speed of analysis as the files were considerably smaller.

3 Density Determination of Homogeneous Materials

To determine the density of an unknown material, a homogeneous polymer sample was scanned together with the calibration set and its density determined according to its average grey value. The so determined density was used to identify the sample. Figure 3 shows from left to right the unknown sample followed by the calibration set of 7 disks. In this case only 7 disks were used, due to space limitations in the cylinder case of the samples (delrin acetal, one of the polymers of intermediate density, was removed). All further work reported here use 7 polymers for calibration.

Using the calibration function determined from the calibration disks, the CT density of the sample was determined

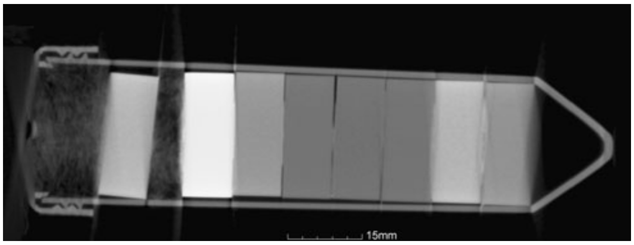

Fig. 3 2D view of the unknown sample (*left*) and the calibration set (*right*)

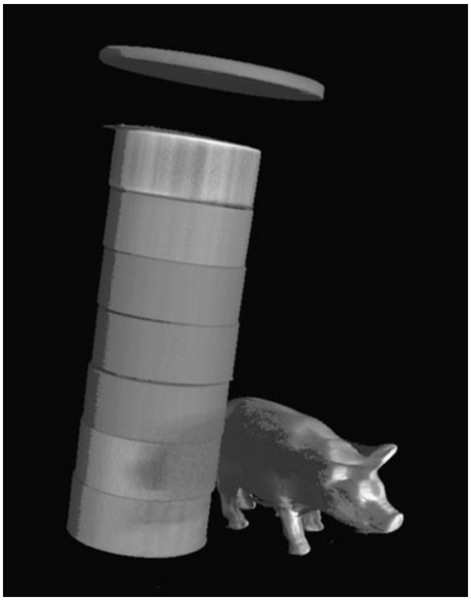

Fig. 4 A plastic disk (*top*) and a toy pig (*right*) of unknown materials, with the calibration stack of polymer disks

to be 1.3 g/cm^3. The experimentally determined density (weighing and physically measuring volume using a calliper and calculating geometrically) was 1.38 g/cm^3 with the difference being 0.08 g/cm^3, or about 6 %. This allowed the identification of the unknown polymer as polyethylene terephthalate (PET).

If the volume of the samples in question is too complex to determine experimentally, it is possible to determine the volume of the 3D object from the CT scan and thereby calculate its mass from the CT determined density and the CT determined volume. It is then easier to compare the CT determined mass with the actual mass, to evaluate the accuracy of the CT method.

To test the CT method with more complex samples, various objects where scanned including a toy disk and a pig (Fig. 4), together with the stack of calibration polymers.

The CT determined density of the toy disk was 1.12 g/cm^3 and the CT determined mass was calculated as 3.02 g. Its actual mass was 2.98 g, which is a deviation of 1.5 % and proves the viability of the CT technique. The toy pig, on the other hand, had a CT determined density of 2.88 g/cm^3 and a calculated CT mass of 16.6 g. Its actual mass, however, was only 8.6 g. This is a significant error of close to 100 % and an explanation for this discrepancy is attempted in the following sections.

4 Sources of Error

Potential error sources in the density determination described above can be either due to artefacts, or due to chemical compositional differences. Since X-ray attenuation is a function of both density and atomic composition [12], a polymer with molecules with a larger atomic number will lead to higher X-ray attenuation and a higher CT density and hence CT mass. The effects of artefacts are not discussed further as there are standard techniques to reduce these, and in our work we found that this is minimized by ensuring high signal to noise ratios, and using enough projection images. We also found that the size of the object can change the density measurement, when beam hardening artefacts are present. Therefore the object under investigation should be roughly the same size as the samples from the calibration set, when in doubt over artefacts causing potential errors. We found that object complexity plays a minimal role. A point to note is that the use of higher X-ray flux improved the signal to noise, resulting in relatively less artefacts. It was also noted that artefacts are always present around the outer edges of the reconstruction volume, though sometimes only visible in the grey value data.

To determine the effect of chemical composition of the polymer, the toy pig, which showed the large deviation between CT determined and actual mass was further cut into a small disk shaped section of roughly the same size as the calibration disks and scanned together with the calibration set, again resulting in a CT mass estimate higher than the actual mass. The simplified shape and size, similar to the calibration disks, indicates that the discrepancy is due to effects other than CT scan artefacts.

The calibration polymers were subsequently scanned together with the toy pig offcut at both 60 kV and 230 kV. The resulting calibration curves for the known polymers at both 60 kV and 230 kV are shown in Fig. 5, both curves having R^2 values of better than 0.99. The reason for the relatively better fit than Fig. 1, is the increased flux (using a direct tube resulting in better signal to noise ratio) and the decreased resolution of these scans (140 µm, resulting in less reconstruction artefacts around the edges).

The average grey value for each calibration disk from scans at 230 kV and at 60 kV were divided by one another and this ratio was normalized to the ratio of one of the calibration disks, providing values in the range from 1.0 to 1.1. However the ratio for the toy pig was 0.67, as shown in Fig. 6. This confirms that the material is of a different

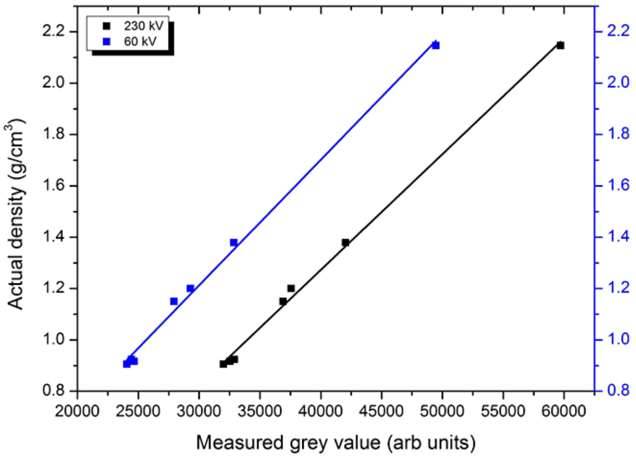

Fig. 5 Calibration data at 60 kV and 230 kV

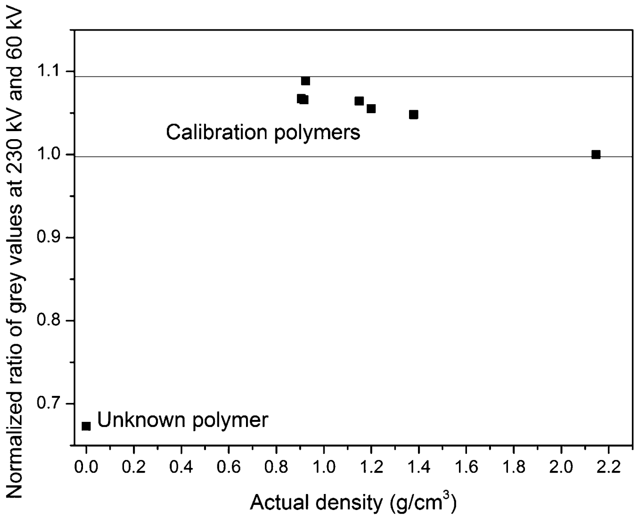

Fig. 6 Dual energy check for unknown polymer (toy pig offcut)

chemical composition, i.e. not a purely organic polymer, than the calibration polymers, but probably contains some heavier atoms. An analysis using a portable XRF instrument showed evidence of chlorine and calcium. Either of these would result in higher CT grey values than expected. Since this material's dual energy ratio does not lie within the range indicated by the calibration samples, the calibration function will not be applicable to this material.

In an example with a different unknown polymer, the ratio for the unknown polymer was found within the range of the calibration as shown in Fig. 7. In this case, the calibration function is applicable, and was used to determine the density of this material as 0.89 g/cm^3 from the 230 kV calibration function and 0.93 g/cm^3 from the 60 kV calibration function. This helped to identify the polymer as medium density polyethylene (expected to be in the range of 0.9–0.94 g/cm^3).

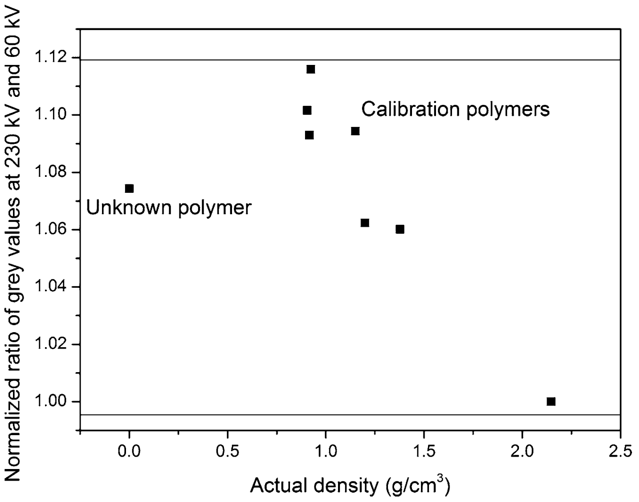

Fig. 7 Dual energy check for unknown polymer, lying within the band of calibration polymer ratios

5 Conclusions

We have demonstrated that microCT scanning allows the quantitative determination of the density (and mass) of a polymeric object. The repeatability and accuracy were demonstrated with a calibration set of polymer disks, from which a fitting function could be derived to calculate the density of an unknown material. The method was found to produce reliable results when the unknown object had the same size and comparable chemical composition as the calibration disks. The main error source was found in the chemical composition, which might result in higher or lower CT density and mass measurements. Dual energy scans were used to identify if an unknown polymer was within the same compositional range as the calibration polymers, therefore providing a test if the calibration function would be applicable. This methodology can help to very accurately quantify densities of polymers and polymer composite materials.

Acknowledgements Stellenbosch University and National Research Foundation funding was used in this project. The CT scanner was granted by the National Research Foundation through the National Equipment Program. We acknowledge useful comments from reviewers in a previous version of this manuscript.

References

1. Mull, R.T.: Mass estimated by computed tomography: physical density from CT numbers. Am. J. Roentgenol. **143**(5), 1101–1104 (1984)
2. Philips, D.H., Lannutti, J.J.: Measuring physical density with X-ray computed tomography. Nondestruct. Test. Eval. Int. **30**(6), 339–350 (1997)
3. Lang, T.F., Li, J., Harris, S.T., Genant, H.K.: Assessment of vertebral bone mineral density using volumetric quantitative CT. J. Comput. Assist. Tomogr. **23**(1), 130–137 (1999)

4. Zou, W., Hunter, N., Swain, M.V.: Application of polychromatic µCT for mineral density determination. J. Dent. Res. **90**(1), 18–30 (2011)
5. Lindgren, L.O.: Medical CAT scanning: X-ray absorption coefficients, CT numbers and their relation to wood density. Wood Sci. Technol. **25**, 341–349 (1991)
6. Freyburger, C., Longuetaud, F., Mothe, F., Constant, T., Leban, J.-M.: Measuring wood density by means of X-ray computer tomography. Ann. For. Sci. **66**, 804 (2009)
7. Macedo, A., Vaz, C.M.P., Pereira, J.C.D., Naime, J.M., Cruvinel, P.E., Crestana, S.: Wood density determination by X- and gamma-ray tomography. Holzforschung **56**, 535–540 (2002)
8. Stoel, B.C., Borman, T.M., de Jongh, R.: Wood densitometry in 17th and 18th century Dutch, German, Austrian and French violins, compared to classical cremonese and modern violins. PLoS ONE **7**(10), e46629 (2012)
9. Meincken, M., du Plessis, A.: Visualising and quantifying thermal degradation of wood by computed tomography. Eur. J. Wood Prod. (2013)
10. Sasov, A.: X-ray microtomography of plastics and composite materials. In: Bonse, U. (ed.) Proceedings of SPIE, Developments in X-Ray Tomography III, vol. 4503, pp. 274–281 (2001)
11. Schilling, P.J., Karedla, B.R., Tatiparthi, A.K., Verges, M.A., Herrington, P.D.: X-ray computed microtomography of internal damage in fiber reinforced polymer matrix composites. Compos. Sci. Technol. **65**, 2071–2078 (2005)
12. Cho, Z.H., Tsai, C.M., Wilson, G.: Study of contrast and modulation mechanisms in X-ray/photon transverse axial transmission tomography. Phys. Med. Biol. **20**(6), 879–889 (1975)