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CALIBRATION OF QUANTITATIVE NEUTRON RADIOGRAPHY METHOD FOR MOISTURE MEASUREMENT

Tomaž Nemec, Robert Jeraj

Reactor Physics Division, "Jožef Stefan" Institute, Jamova 39, 1111 Ljubljana, Slovenia

e-mail: tomaz.nemec@ijs.si

Abstract

Quantitative measurements of moisture and hydrogenous matter in building materials by neutron radiography (NR) are regularly performed at TRIGA Mark II research reactor of "Jožef Stefan" Institute in Ljubljana. Calibration of quantitative method is performed using standard brick samples with known moisture content and also with a secondary standard, plexiglas step wedge. In general, the contribution of scattered neutrons to the neutron image is not determined explicitly what introduces an error to the measured signal. Influence of scattered neutrons is significant in regions with high gradients of moisture concentrations, where the build up of scattered neutrons causes distortion of the moisture concentration profile. In this paper detailed analysis of validity of our calibration method for different geometrical parameters is presented. The error in the measured hydrogen concentration is evaluated by an experiment and compared with results obtained by Monte Carlo calculation with computer code MCNP 4B. Optimal conditions are determined for quantitative moisture measurements in order to minimize the error due to scattered neutrons. The method is tested on concrete samples with high moisture content.

1. INTRODUCTION

Quantitative measurement of moisture in porous building materials is important for civil engineering and building materials industry. Qualitative detection of moisture by neutron radiography (NR) is well established. For quantitative measurements recorded neutron image needs to be corrected for various contributions of neutron beam components. Quantitative method is calibrated by standards with known moisture content. Using the calibration curve we can convert the corrected signal into hydrogen concentration values [1]. Influence of scattered neutrons to the image formation has to be determined and subtracted from measured signal to obtain the signal of transmitted neutron beam.

Quantitative measurements of moisture and hydrogenous matter in building materials are regularly performed at TRIGA Mark II research reactor of "Jožef Stefan" Institute. Our quantitative method is calibrated with standard samples with known moisture content and with a secondary standard, plexiglas step wedge. Contribution of scattered neutrons to the neutron image is not measured explicitly but is taken into account through the calibration curve. This simplification introduces an error to the measurements due to signal of scattered neutrons. This error is low enough for thin samples at low moisture concentrations while at increased concentrations of moisture the scattered component prevails over the transmitted component and then the calibration is not correct. Influence of scattered neutrons to the image is significant in the regions with high gradients of moisture concentrations, where build up of scattered neutrons causes distortion of the moisture concentration profile [2].

In this paper a detailed analysis of validity of our calibration method for different geometrical parameters is presented. Optimal conditions for quantitative measurements are determined by an experiment. The error in the concentration profiles at regions with high gradients is estimated by the experiment and also by Monte Carlo calculation. Improved

method with minimized error due to scattered neutrons is tested on concrete samples with high moisture content.

2. QUANTITATIVE METHOD FOR MOISTURE DETECTION

For quantitative determination of the moisture content at position (x, y) in a thin sample of well defined geometry and thickness d, the attenuation of a collimated neutron beam Φ/Φ_0 is ideally correlated with the mass concentration C_w of water:

$$\Phi(x,y) = \Phi_0(x,y) \exp[- (\mu_m + \mu_w C_w(x,y)) \rho_m d] \quad (1),$$

where μ_m and μ_w are the neutron macroscopic mass attenuation coefficients of the material and water, respectively. Using a detector with linear dependence of observed detector signal on the neutron exposure dose [3] we get:

$$S = k \Phi t = k \Phi_0 t \exp[- (\mu_m + \mu_w C_w) \rho_m d] \quad (2),$$

where S is the detector signal at certain coordinates (in units PSL mm^{-2} for imaging plate detectors), k is the detector sensitivity and t is the exposure time. However, in a real case, the recorded signal S' includes also the background due to gamma-rays, epithermal neutrons and neutrons scattered in the sample (S_γ, S_{ep} and S_s , respectively):

$$S' = S + S_\gamma + S_{ep} + S_s \quad (3).$$

Background components need to be subtracted from recorded signal to obtain S and then log S is proportional to the moisture concentration C_w. The effects of the background due to γ-rays and epithermal neutrons can be estimated by imaging neutron beam, where thermal neutrons are filtered out with a filter made of boral and gadolinium plates. Results of the measurements need to be corrected for neutron beam nonhomogeneities. The effect of scattered neutrons, primarily due to scattering on hydrogen, increases with the mass thickness of hydrogen. However, the influence of scattered neutrons on contrast becomes small enough at increased distance between the sample and the detector [4].

2.1. Calibration of quantitative method

Calibration is performed with a series of standard samples with known moisture content (in the range 1 to 14 wt % for red brick samples). NR image of the standards is performed in the same experimental conditions as actual measurements. The homogeneity of moisture distribution in the standards is checked by NR and is varying by less than 0.5 wt %. Standards are enveloped in Al foil to prevent moisture evaporation and the weight control during 1-2 days shows that weight loss of samples is negligible. For routine measurements a secondary standard is used, plexiglas step wedge (0.5 to 5 mm thick, 0.5 mm steps). Standard step wedge is intercalibrated with the set of standard building material samples.

2.2. Contribution to the NR image of neutrons, scattered in the sample

Contribution of scattered neutrons to the neutron image is not measured explicitly but is taken into account through the calibration curve. This simplification introduces an error to the measurements because signal of scattered neutrons is added to the signal of transmitted neutrons and detection sensitivity is decreased. This error is negligible small (compared to contribution of background) for thin samples at low moisture concentrations (1-10 wt % H_2O for 2 to 2.5 cm thick red brick samples). However, at increased moisture concentration the scattered component can prevail over the component of transmitted thermal neutrons and in such conditions the calibration is not correct. Influence of scattered neutrons to the image is important in regions with high gradients of moisture concentrations, e.g. at the front of advancing water in porous sample where build up of scattered neutrons causes the distortion of the moisture concentration profile. This effect is significant also at low enough moisture

concentration where our calibration procedure is valid. To diminish the contribution of scattered neutrons to the NR image appropriate distance between the samples and the neutron imaging detector must be taken.

3. EXPERIMENTAL

3.1. Neutron radiography using neutron sensitive imaging plates

Neutron radiographic examinations of samples are performed in the NR facility in the thermal column of the TRIGA Mark II research reactor in Ljubljana. Characteristics of neutron source were described in the literature [1, 5]. For neutron imaging FUJI imaging plate neutron detector (IP-ND), which is very suitable for quantitative measurements because of the linear response in wide exposure range, good spatial resolution and high sensitivity, is used. Characteristics of IP-NDs are presented in literature [1, 3]. Exposure times at thermal neutron flux of $4 \cdot 10^5$ cm^{-2} s^{-1} ranged from 1 to 60 s. Contribution of gamma ray and epithermal neutrons to the NR image was found to be up to 14 % of the total signal in the neutron beam. The statistical error in the detector signal ranged from 1.5 to 3 %.

3.2. Monte Carlo simulation

Monte Carlo (MC) simulations of NR experiments are performed using MCNP 4B computer code [6]. A parallel neutron source of the same size as the cross-section area of the investigated object was defined, with the counting resolution of 1 mm. The source with Maxwell type spectrum with E=0.025 eV was assumed. Statistical error was below 0.5 % for primary neutron and increases to a few % for neutrons scattered in the area around the sample. Neutron contributions to the NR image were determined separately as primary (undisturbed or transmitted) and total neutron flux which is the sum of the primary and neutrons scattered in the sample. Detected signal was normalized to 1 source particle.

3.3. Investigated samples and standards

Calibration with standards was performed in investigation of moisture transport in red brick samples [7]. Standards for calibration were prepared from same material and with the same thickness and moisture concentrations as investigated samples. Standard step wedge was cut from commercial plexiglas. Concrete samples were prepared according to standard procedure for concrete impregnation tests. Samples for MC simulations were defined with the same dimensions as the samples investigated in NR experiments. Composition of plexiglas and concrete was taken from literature and samples were assumed to be uniform in the volume.

4. RESULTS AND DISCUSSION

4.1. Error in the calibration curve due to contribution of scattered neutrons

By calibration curve the detector response was correlated with hydrogen concentration in standards. Calibration curve with standards of known moisture content is shown in Fig. 1a and calibration with plexiglas step wedge in Fig. 1b. Neutron attenuation profiles (measured in NR images) were corrected for contribution of background. In Fig. 1c calibration curves for plexiglas step wedge obtained by MC simulation are shown. Object-detector distance in Fig. 1a varied from 0 to 10 cm, while in Fig. 1b and in Fig. 1c this distance was 1 cm. From calibration curves in Fig. 1a it is seen that effect of scattered neutrons build-up increases with approaching of samples and detector. At distance 10 cm linearity exists up to 10 wt % H_2O but at higher concentrations the error in calibration due to scattering is noticed. At short distances the calibration curves are not linear. The calibration curve in Fig. 1b is linear from 0.5 to 4.5 mm thick plexiglas at only 1 cm from the detector. In Fig. 1c (MC simulation) linearity is observed both for transmitted (direct) component and also for the total neutron

flux. From Figs. 1b and 1c we can see that calibration is valid in limited range of hydrogen mass thickness even with scattered component present in NR image. Calibration is valid only at specific experimental conditions which need to be determined separately for every type of scattering samples and precise experimental geometry.

4.2. Resolution degradation due to scattering in area of high gradients of H mass thickness

NR and MC simulation images of plexiglas step wedge covered in part by Gd filter are presented in Fig. 2 together with the measured profiles of neutron attenuation along the wedge and below the Gd filter. The signal recorded by the detector below the Gd filter in NR image (Fig. 2a) is caused by neutrons scattered in the plexiglas wedge. Profile of sharp edge of Gd is distorted due to scattered neutrons. For comparison a MC simulation was performed in the same geometry as the NR experiment and signals due to transmitted and scattered neutrons were distinguished. Results of MC simulation in Fig. 2b are very similar (distribution of scattered neutrons below the Gd filter) to the NR measurements. When object-detector distance is increased this distorting scattering effect vanishes but at same time NR image resolution is worsened by the geometrical unsharpness caused by divergence of neutron beam. Geometrical unsharpness is not seen in MC simulation where parallel neutron source was defined.

4.3. Comparison of NR experiment and MC simulation

In Fig. 3 images by NR (a) and MC simulation (b) imaging of sharp moisture front in a concrete sample (7.1 by 3.5 cm, 1.4 cm thick) placed 10 cm from the detector are seen. Measurement of moisture in concrete is difficult due to nonhomogeneity of material and high content of bound water in concrete. The MC image profiles taken across the border of wet and dry areas are shown in Fig. 3c. At this distance from detector the scattering background is below 10% of total signal and is uniform so we can subtract it from the image. Resolution degradation is not significant.

This experiment shows the clear advantage of using MC simulation as complementary method to NR experiments. By MC we distinguish between transmitted and scattered neutron components and obtain results at better contrast with subtraction of scattered component from the image. These MC simulations were ideal cases and did not match exactly the real conditions at NR imaging. Shape of neutron spectrum was not determined and by assuming a Maxwell type spectrum results differ from the NR measurements. Other simplifications include the assumed homogeneous composition of investigated materials and their elemental compositions, which were not determined by elemental analysis.

5. CONCLUSIONS

Results presented in the paper clearly illustrate that neutrons, scattered in a hydrogenous sample, cause the degradation of contrast and spatial resolution of neutron radiographic image. Contribution of scattered neutrons may not be neglected in quantitative measurements and it must be determined separately to estimate the error in measured H concentration values due to scattering component. One way to lower the influence of neutron scattering on image contrast is by increasing appropriately the object-detector distance. When calibration is performed with standards placed several cm from the detector the detected signal is linear with the moisture concentration in wide range but with object and detector in contact, the scattering component can be greater than the transmitted component making impossible the H measurement. By MC simulations we found that calibration is valid in limited range even when scattered neutrons are present in the image what allows H measurement without the elimination of scattered neutrons from the NR image. In areas with sharp gradients of H content the resolution is degraded. By increasing the object-detector distance the distortion by

scattering will decrease but the image resolution is worsened by geometrical unsharpness. As compromise, an optimal distance has to be determined for every specific hydrogenous sample.

Investigation of scattering in standard hydrogenous objects by NR and MC simulations showed that in specific experimental conditions the calibration with standard samples and plexiglas step wedge enables quantitative measurements of H concentration in porous building material samples. Scattering and background contributions limit the sensitivity of the measurements. This is a problem when concrete samples with high moisture content and nonhomogeneous structure are examined. So far NR imaging of concrete was limited to qualitative or semiquantitative measurements with low resolution. Future work will be to develop the method to enable NR imaging by subtracting the scattered component what will increase very much the sensitivity of H detection in concrete and enable quantitative measurements. MC was found to be a very useful tool in evaluation of the NR results and will be used in future for simulation of other NR experiments.

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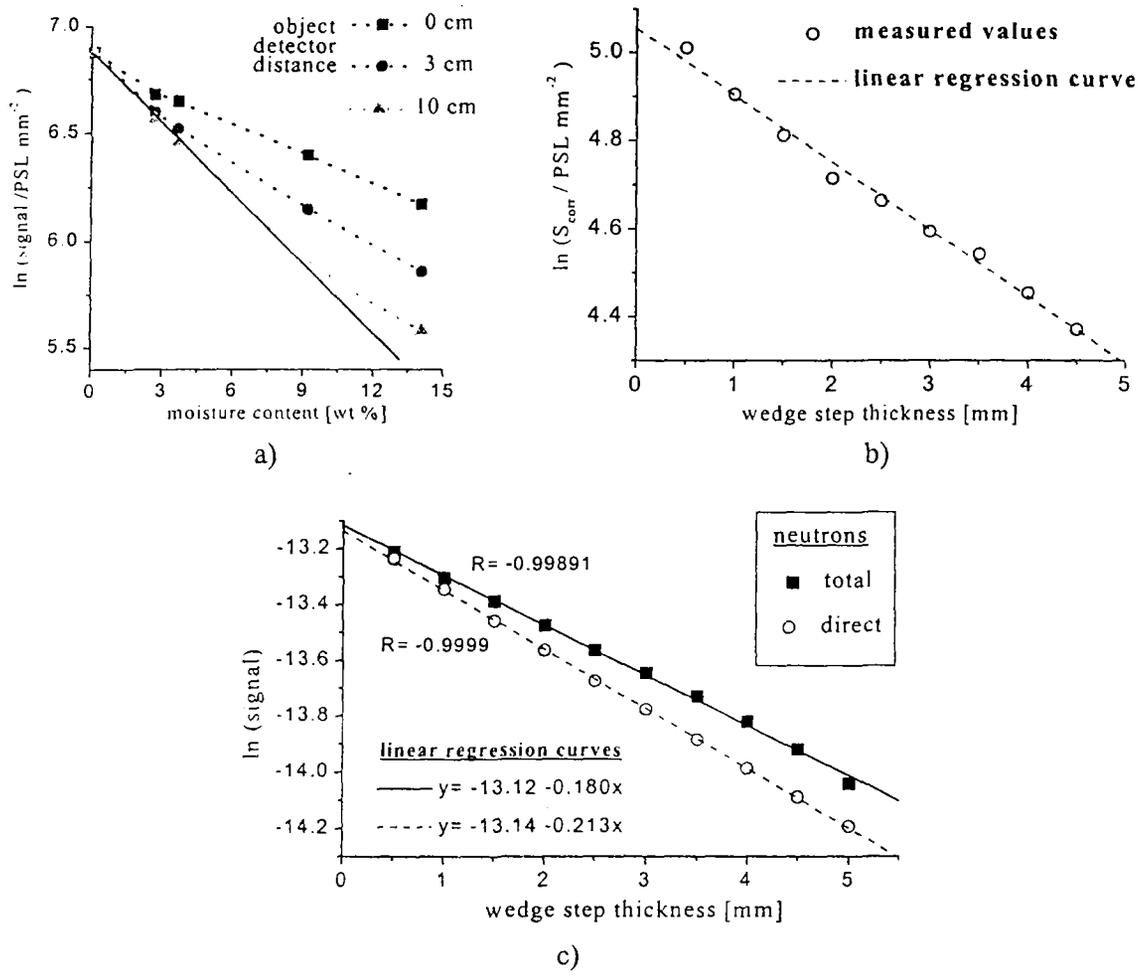
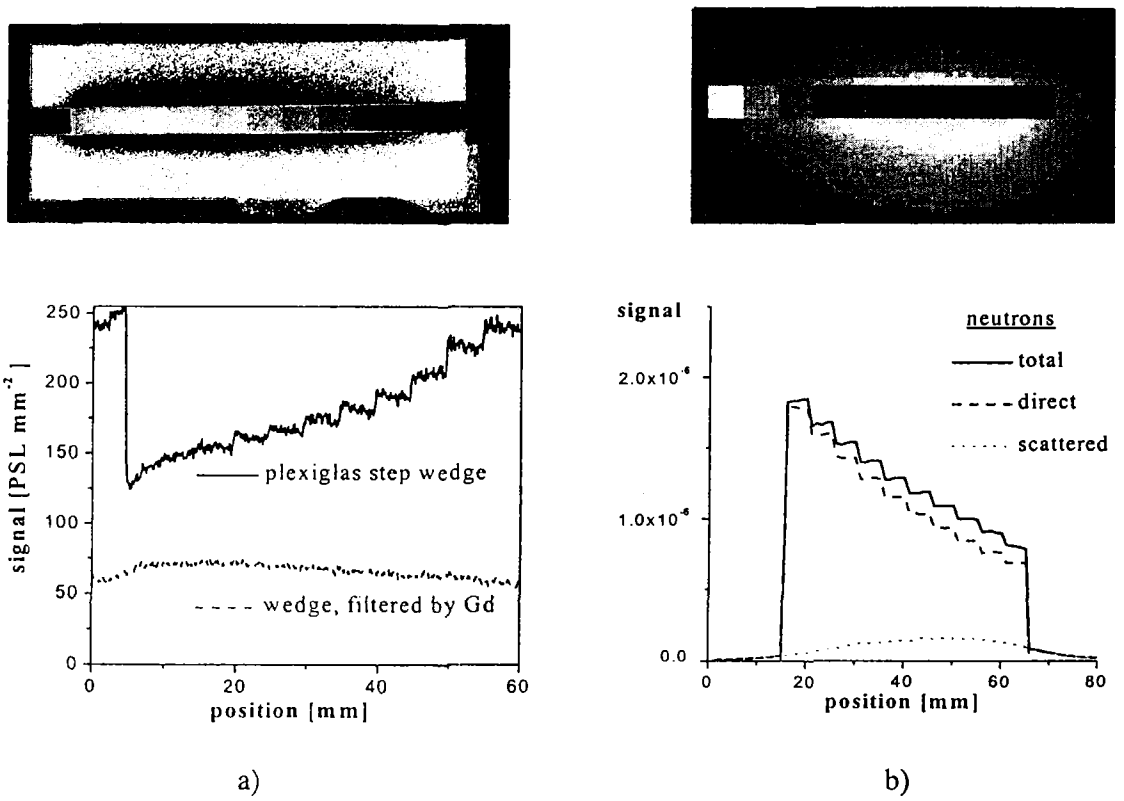


Figure 1. Calibration curves used to determine the correlation between detector response and hydrogen concentration. Calibration by red brick standards with known moisture content (a), measured at varying distance from IP-ND. Calibration with standard plexiglas step wedge 0.5-5 mm thick placed 1 cm from detector, obtained by NR (b) and by MCNP simulation (c).



a) b)

Figure 2. Degradation of neutron image resolution due to scattering in areas with high gradients of H concentration. The object is plexiglas step wedge which is filtered by 100 μm thick Gd stripe in upper part: (a) NR image and neutron attenuation profiles along the wedge and below single Gd (lower part of wedge is covered by two Gd stripes), b) image by MCNP simulation. Object-detector distance is 1 cm. Below are shown: (a) the neutron attenuation profiles and (b) contributions of individual components calculated by MCNP (around the object the signal is caused only by scattered neutrons).

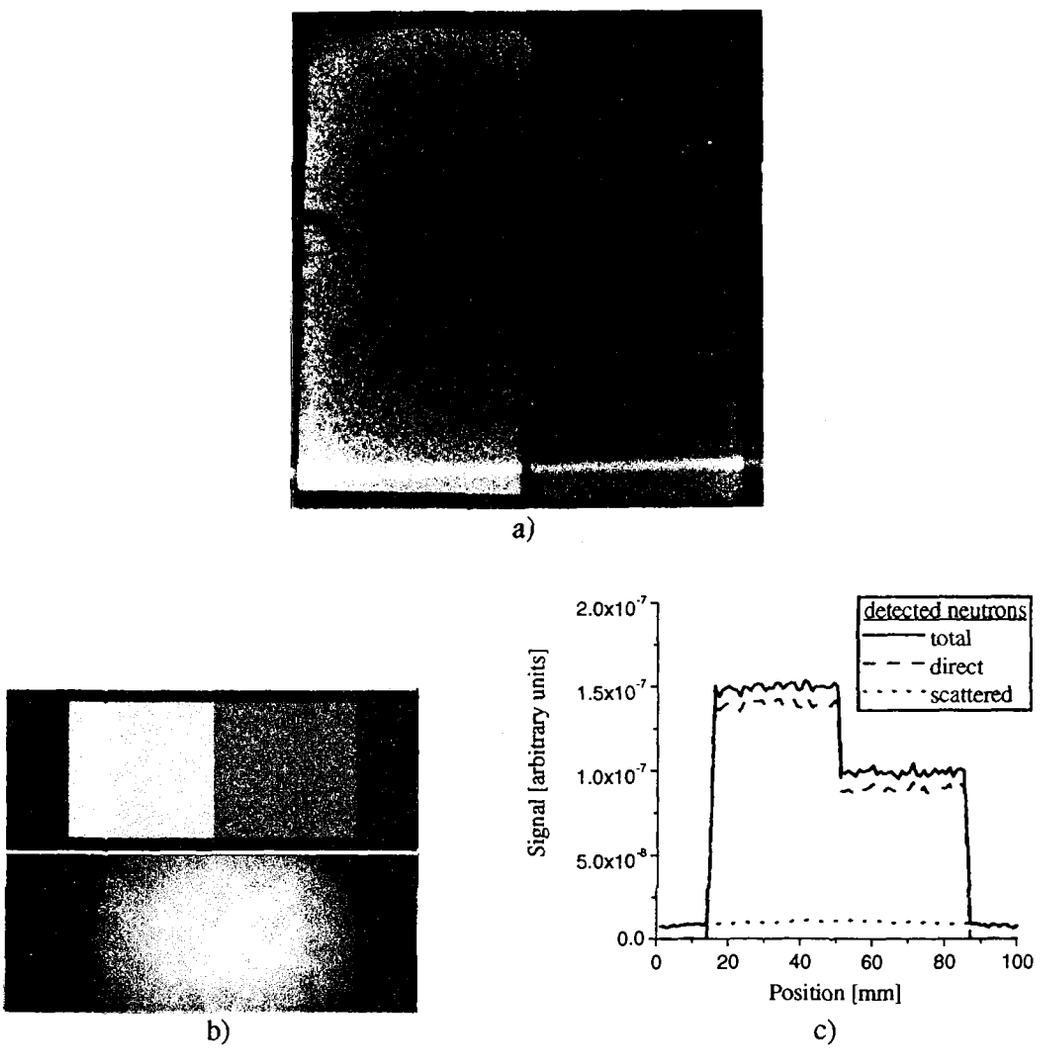


Figure 3. NR and MCNP images of wet concrete sample at object-detector distances 10 cm, contrast is increased: a) NR image of wet and dry concrete samples in close contact to simulate sharp edge of water front, b) MCNP image of concrete sample wet to 1 half, in c) are shown profiles of total and scattered contributions. Profiles are noisy due to statistical error.