Particle moisture distribution during drying and storage of grain analyzed by TD-NMR spectroscopy

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Abstract

In grain preservation and storage, the term "grain moisture content" refers to the average moisture content of a sample of kernels. Besides it, however, the known variation of the single kernel moisture should be considered to evaluate post-harvest processes like drying and storage. The aim of this work was to adjust the Time Domain Nuclear Magnetic Resonance method for measuring the single kernel moisture content and to test the applicability of this method to investigate the dynamics of the particle moisture distribution (PMD) during grain drying and storage. For this purpose, basic laboratory-scale experiments were conducted at first in a drying chamber and small storage containers, respectively, using storable wheat as test material. The drying experiments revealed that, as the drying time and oven temperature were increased, the width and the standard deviation of the PMD gradually increased. This tendency inverted, however, as soon as the drying temperature and time exceeded certain limits. As the storage experiments revealed, the width of the PMD noticeable decreased just when there was enough air in the container available for moisture exchange. Thereafter, the storage experiments were extended to pilot scale using barrels to store the grain for months. These experiments were performed without ventilation so as to simulate dead zones of airflow that often occur under industrialscale flat store conditions. In closed barrels, no differences were observed in the dynamics between storable farm-fresh and dried wheat. There was no equalization of the particle moisture even after seven months of storage.

Keywords: grain; TD-NMR spectroscopy; single kernel moisture; moisture distribution; post-harvest processes

1 Introduction

Studies have found that a significant variance in the moisture content exists among individual grain kernels (Liu, Montross, & Bakker-Arkema, 1997). The water content of a sample of grain may be determined with some precision. However, since the individual kernels vary in shape, size, consistency, and degree of maturity at harvest, it is reasonable to expect differences in their water content, even though they have lain together in the same container for a considerable period of time (Oxley, 1948). Thus, the variation in the moisture content should be considered in addition to the average moisture content when evaluating the processes of grain drying and storage (Liu et al., 1997). The moisture content of grains is one of the most important parameters in measuring quality and predicting storability. However common meters are unable to detect any differences in moisture distribution is, however, important for better prediction of grain storability. The non-uniformity of the moisture distribution within a grain mass is known to affect its storability (Brusewitz, 1987). Furthermore, measuring the moisture distribution of single kernels provides an indirect method for examining the exchange of water between single kernels and the atmosphere (Oxley, 1948).

Oxley (1948) was among the first to measure the moisture content distribution of individual wheat kernels. Using the oven method, he examined the particle moisture distribution after harvest, storage and drying. The grain was stored in small containers (sealed 200 ml glasses and 30 ml tubes) for periods of one month to one year. During the storage, the standard deviation of moisture content decreased. Likewise, Montross, Bakker-Arkema, & Hines (1999) examined the moisture distribution of maize after harvesting, drying and 200 hours of storage in sealed plastic bags. As they could show, the moisture distribution of the maize kernels became narrower during storage. Brusewitz (1987) recognized that grain samples of different moisture contents that were mixed together tended to approach over a period of time, but did not reach equilibrium at an intermediate moisture content. In another study, Henderson (1987) stored barley dried down to 7 % and barley moistened up to 23 % in a sealed container. He revealed that 90 % of the moisture exchange took place within 3 days. However, it should be noted that wetted grain does not act like naturally moist grain because of different moisture bonds and moisture distribution (Schreiber, Mühlbauer, Wassermann, & Kuppinger, 1981). Even after

several hours of rewetting, the moisture resided only in the outer regions of the kernel but not in the center (Fortes, Okos, & Barrett, 1981).

There are two reasons for fluctuations of the single kernel moisture content:

(1) Grain kernels may vary physically such that their water-holding capacities differ from each other when in equilibrium with a given atmospheric relative humidity.

(2) The kernels may be in equilibrium with different relative atmospheric humidities (unsteady state). The kernels exert different water vapor pressures, and if such kernels are contained in a common container, they will exchange water by diffusion until all kernels exert the same vapor pressure. They will then be in equilibrium with the same relative humidity and the remaining differences in water content will be due to reason (1) above. The time required for such equalization depends strongly on the size of the container (Oxley, 1948).

The Time Domain Nuclear Magnetic Resonance (TD-NMR) was selected and adopted as a method for a fast and non-destructive measurement of the single-kernel moisture content. This method has already been applied by Peglow, Cunäus, & Tsotsas (2011) to determine the mass of water in a single solid particle to investigate the distributed properties of a population of granular materials after drying in fluidized bed dryers.

Postharvest processes for grain influence the particle moisture content distribution depending on the particular process conditions. It can be expected that, the more homogeneous the PMD is after drying, the higher will be the process quality of subsequent storage. If the moisture content is widely distributed, it can be assumed that there are still comparatively wet kernels which can create moisture accumulates during storage. These moisture accumulates can be the initial point for mould infestation. Therefore, the variation in the particle moisture content should be considered in the process evaluation besides the average moisture content.

Besides the method development, the aim of the work was to demonstrate the applicability of the TD-NMR for studying the dynamics of the particle moisture distribution during post-harvest processes such as drying and storage of grain. For this purpose, basic laboratory-scale experiments were conducted at first in a drying chamber and small storage containers (micro test tubes, glass test tube, and plastic container), respectively, using wheat as test material. The aim was to show the influence of process parameters on the PMD by varying the drying temperature, drying time, the volume of the storage container and the storage time. In a second test series, the storage experiments were extended to pilot scale using 120-litre-barrels to store the grain for months without ventilation. These experiments were conducted without ventilation so as to simulate dead zones of airflow in real industrial-scale flat stores which can occur due to inhomogeneous airflow conditions. The experiments were performed using storable farm-fresh as well as dried wheat as test materials. The aim was to investigate the effects of exchange and equalization of particle moisture during long-term storage.

2 Materials and methods

2.1 Materials

For the laboratory drying and storage experiments, wheat of one batch has been used that was stored before for several months under cold conditions (10 $^{\circ}$ C). The harvesting moisture was between 18 and 19 % wb. All grain moisture contents in this article refer to wet basis (wb).

The pilot scale long-term storage experiments were conducted using one batch of farm-fresh premium-wheat ("Discus") with a harvesting moisture of around 14,8 % wb and a second batch of farm-fresh superior-wheat with a harvest moisture of 16,2 %. Before storage, the second batch was dried in a mixed-flow dryer (at an air temperature of 60 °C) down to an average moisture content of 14,7 %.

2.2 TD-NMR spectroscopy

To determine the single grain moisture content, a Time-Domain NMR Benchtop System Minispec mq40 (Bruker) was applied. This system is recommended by the producer for single kernel measurements. The function of the TD-NMR and the Hahn-echo pulse sequence used has been explained elsewhere, see for example (Cunäus, 2010; Hahn, 1950; Peglow et al., 2009; Rutledge, 2001; Todt, Guthausen, Burk, Schmalbein, & Kamlowski, 2006).

With the NMR spectroscopy, the water content can be determined only indirectly. Therefore, a calibration is required with a direct technique. Since the NMR spectroscopy is a non-destructive measuring method that does not change the properties of the sample, it is possible to determine the moisture content of a kernel after the NMR measurement, e.g. by means of the oven method (ASAE S352.2 FEB03), and to calibrate the NMR with these results. For each batch of grain a separate calibration is necessary.

According to the ASAE standard, replicate determinations using 10 g of unground grain each should check within 0.2 % wb of moisture. Instead of the prescribed 10 g samples, however, only single kernels could be weighed out. As a consequence, double determinations were not possible in that case. Nevertheless, the ASAE standard using the oven method was suitable for the calibration due to its low susceptibility to errors and the high number of kernels that can be used for calibration.



Fig 1: Angle template to adjust the grain in the TD-NMR device. Each measurement was performed in the kernel position as shown in figure (left). Particle moisture distribution as a function of the kernel angular position 1-8 (right) measured for wheat with 14.7 % wb (mean values from n=30 kernels).

Own pretests with the TD-NMR method conducted for different types of grain (wheat, barley, rye, and oats) revealed a strong influence of the shape of the particle and its position on the NMR moisture signal. As the kernels were turned by 90°, the moisture deviated by up to 0.94 % wb (for wheat), and this difference increased as the grain water content increased, see Fig. 1. This effect has already been reported by Tiwari, Gambhir, & Rajan (1974) as well as Srinivasan (1979) while measuring the oil content of single seeds of maize, sunflowers, soya, and groundnut. In their studies, the transient wide-line NMR technique was used. This means, for elongate shaped particles such as wheat grains, the kernel angular position in the NMR spectrometer must be considered while measuring its moisture content. As a consequence, for the calibration of this measuring method, the kernels were always placed in the same angular position in the NMR spectroscope (Fig. 1a). The subsequent measurements were then performed in the same kernel position.

2.3 Laboratory drying and storage experiments

The laboratory drying experiments were performed in a drying chamber (type FED 115, Binder). To determine the moisture distribution during drying, samples of 100 kernels were selected from the same batch of wheat and were then dried in the oven. The kernels were placed on top of a net in the oven without any contact to each other so as to assure convective heat transfer without heat conduction. The kernels were dried at 70, 100 and 130°C for 5, 10 and 30 min each. After drying, the kernels were cooled down at room temperature in a desiccator for one hour and were then measured using NMR spectroscopy (always in the same angular position).

Table 1: Storage containers and available air volume per kernel			
Container volume Air volume per kernel			
0.2 cm ³ (micro test tube)	0.16 cm ³		
10 cm ³ (test tube)	0.06 cm ³		
100 cm ³ (plastic container)	0.96 cm ³		



Fig. 2: Storage containers: micro test tube with one kernel (left), test tube with 100 kernels (middle) and large plastic container with 100 kernels (right).

The particle moisture distribution of a grain bulk which has been dried before influences the downstream process of storage. Therefore, the dynamics of the PMD of a wheat sample were investigated under laboratory conditions during storage after a prior drying process. The wheat sample was dried on top of a net in the oven at 100 °C for 10 min. Thereafter, it was cooled down at room temperature for 1h in a desiccator, and then the moisture content was determined with the NMR-spectrometer.

In order to study the influence of the volume of the storage container on the PMD over the time of storage, micro test tubes, a glass test tube, and a plastic container were used as laboratory storage containers, see Table 1 and Fig. 2. The samples were stored at a temperature of 8 °C. One sample of 100 kernels was stored in individual micro test tubes (0.2 ml), 100 kernels together in a glass test tube (10 ml), and 100 kernels together in a sealed large plastic container (100 ml). The storage time was 14 and 56 days. Before each NMR measurement, the grain kernels were acclimatized in the closed containers for one hour at room temperature in each case. Table 1 shows the different container volumes with the corresponding specific air volume per kernel. To determine the specific air volume per kernel, an average kernel volume of 0.038 cm^3 was considered.

2.4 Pilot-scale storage experiments

The pilot-scale storage experiments were performed using 120-litre-barrels which were completely filled with grain at the beginning, see Fig. 3. The barrels were not ventilated. For the experiments, both farm-fresh and dried wheat were used as test materials. One batch of the farm-fresh wheat (14,8 %) was stored in open barrels (A), one batch in closed barrels (B), see Table 2. Another batch of wheat, that was harvested with 16,2 % wb of moisture, was dried down to an average moisture content of 14,7 % in a mixed-flow dryer. This batch was stored in closed barrels (C). For reproducibility, each experiment was conducted in three barrels in parallel. The grain was stored at room temperature with an average of 19,3 °C (\pm 1,09°C standard deviation) for seven months. Samples were taken in regular intervals (19 days, 35 days, 56 days, 56 days, 56 days) by means of a sample insertion device. The samples were drawn from above at two different heights (Fig. 3). The caps of the closed barrels were opened for this purpose. After it, the moisture distribution of 50 kernels of each sample was measured in the TD-NMR.

Table 2: Pilot-scale storage experiments

Experiment	Wheat sample	Barrel
No.		open/closed
А	farm-fresh, 14.8 % wb	open
В	farm-fresh, 14.8 % wb	closed
С	dried, 14.7 % wb	closed



Fig. 3: Schematic representation of sampling of the long-stored wheat in barrels

3 Results and Discussions

3.1 Variation of the particle moisture distribution during laboratory drying tests

Before the drying experiments, the initial particle moisture distribution was determined for the original wheat sample, see chapter 2.1. For this, 100 single kernels were randomly selected from the batch and measured using the NMR spectroscope. The moisture distribution is shown in Fig. 4.



Fig. 4: Initial particle moisture distribution of the original wheat sample. 100 randomly selected kernels. Class width of 0.25 moisture-%.

It is depicted as the frequency or number of particles over the moisture content appearing in the respective moisture classes, each 0.25 moisture% in width. As can be seen from the diagram, the initial PMD was approximately normally distributed. It is known that the scattering of the moisture content of farm-fresh or naturally moist grain is caused by inhomogeneities of the kernels due to location factors such as soil conditions, water availability, solar radiation, etc.

The results of the drying tests are summarized in Table 3 where the average moisture content and the standard deviation of the PMD are shown. For all drying experiments, the measured PMD's were nearly normally distributed. As the drying time was increased, the moisture content expectedly decreased and the width (standard derivation) of the distribution increased. This trend could be observed also for higher temperatures. At 130°C, however, the standard deviation decreased after 30 min. In this case the heat impact was probably too high, thereby damaging the kernels. The moisture content of these kernels was as low as 6 % wb.

In contrast to this, Liu et al., (1997) quoted that after thin-layer drying of maize, the kernel moisture distribution attained a smaller standard deviation than before. The authors stated that this is because the kernels of lower moisture content dry more slowly than kernels of average moisture and kernels of higher than average moisture content dry faster. This seems to be logical, but because grain kernels are not uniform particles and can differ physically, they have different water holding capacities.

Drying temperature	Drying time	Mean moisture	Standard deviation in
		content in %	%
initial moisture	-	19.2	0.33
70 °C	5 min	16.5	0.59
	10 min	15.4	0.60
	30 min	12.6	0.66
100 °C	5 min	15.1	0.62
	10 min	13.4	0.58
	30 min	9.6	0.74
130 °C	5 min	13.0	0.69
	10 min	10.6	0.95
	30 min	6.3	0.71

Table 3: Results of the drying experiments: mean value and standard deviation of the PMD

3.2 Dynamics of the particle moisture distribution during laboratory storage experiments

The moisture distribution of a grain bulk existing after drying influences the subsequent storage process. In this study, the dynamics of the particle moisture distribution were investigated for a dried wheat bulk during storage. The wheat batch was dried at a temperature of 100° C for duration of 10 min. The storage experiments were performed under laboratory conditions in small containers. The study aimed to investigate the influence of the storage container volume and the storage time on the PMD. In each experiment, 100 wheat kernels were randomly selected from the batch immediately after drying (after 1 h cooling down in the desiccator = day 0) and stored in the respective containers (Fig. 2). The PMD was then analyzed at day 0, after 14 days, and after 56 days of storage, respectively.

The results are shown in Fig. 5. Fig. 5a shows the particle moisture distribution of 100 kernels which were stored separately in micro test tubes. As the figure illustrates, the moisture distribution remained almost constant over the storage time. This is due to the fact that no moisture exchange was possible between the kernels. On the other hand, just a limited air quantity of 0.16 cm^3 per kernel was available for moisture exchange in the storage container (Table 1). Hence, the kernels stored in micro test tubes could hardly change their moisture content. It should be noted that the analysis was always conducted with the same kernel. This means that each micro test tube was opened on days 1, 2, 5, 14 und 56 and the kernels were exposed to a 40°C atmosphere in the NMR unit for approximately one minute each time. In Fig. 5, only a selection of the results (day 0, day 14 and day 56) is presented.

The results of the storage experiment in the glass test tube (10 cm³) with very little air are shown in Fig. 5b. As the figure illustrates, the PMD became narrower after 14 days and shifted to the left; thus the kernels dehydrated. After 56 days, the distribution did not become narrower, but the normal distribution changed into a bi-modal distribution. This effect was probably caused by a moisture exchange of the kernels in the upper part of the tube with the air on top (Fig. 2). As a result, these kernels attained a lower moisture content expressed by the second peak on the left side of the curve. By contrast, the kernels at the bottom of the tube were truncated from this moisture exchange. The storage experiment in the large 100 ml container resulted in a significant narrowing of

the particle moisture distribution, as can be seen in Fig. 5c. In this case, a comparatively large portion of air was available for the moisture exchange in the container (0.96 cm^3 air per kernel, Table 1). As a result, the mean moisture content was reduced from 13.3 % wb at day 0 to about 11.6 % wb after 56 days. The standard deviation decreased from 0.59 % to 0.36 %.



Fig. 5: Particle moisture distribution of 100 wheat kernels during storage in micro test tubes (0.2 cm^3) (a), in a sealed glass test tube (10 cm^3) (b), and in a sealed plastic container (100 cm^3) (c); measurements at day 0, after 14 days, and after 56 days; class width of 0.25 moisture-%.

3.3 Dynamics of the particle moisture distribution during pilot-scale storage

The farm-fresh wheat (14,8 %) and the dried wheat (14,7 %) were stored in open and closed barrels (Table 2), respectively, without ventilation from August 2015 until March 2016. The PMD was evaluated every eight weeks. In Fig. 6, only the results from the first measurement (19 days after harvest) and the last measurement are depicted in summary. Each curve represents the particle moisture distribution of 150 wheat kernels (50 kernels each from the 3 barrels of one experimental set-up).

As can be seen from Fig. 6a, the PMD of farm-fresh wheat measured in the open barrels (A) at the upper sampling position was getting wider and the kernels were getting dryer from 14,1 % average moisture content to 12,8 % after seven months of storage. A drying effect was also detected at the lower sampling position due to a slowly moving drying front. In contrast to the laboratory experiment in the plastic container (Fig. 5c), where the moisture distribution became narrower over time, the PMD determined at the upper sampling position in the barrels (A) became wider during the storage dehydration process. This is due to the fact that the wheat in the barrels was not dried before storage. In a second test series, farm-fresh wheat of 14.8 % wb was stored in closed barrels (B), see Table 2. The results are shown in Fig. 6b. As can be seen from the graphs, the PMD measured at

both sampling positions remained constant over time. Both, the average moisture content and the standard derivation did not vary even after seven months. This observation is in line with the results from Montross et al., 1999 (for Maize) and Oxley, 1948 (for wheat).

For comparison with the test series (B), a batch of dried wheat of 14.7 % wb was stored in closed barrels (C) as well. The results are depicted in Fig. 6c. As the figure illustrates, the particle moisture distribution remained nearly constant over time at both positions. As compared to experiment (B), the standard derivation of the PMD was higher. This is because the wheat in experiments (C) has been dried prior to the storage. In principal, the results from experiment (C) correspond to those from the laboratory experiments in the glass test tube (Fig. 5b). However, in the barrels there was much less air available for moisture exchange, hence, there was no change in the moisture content.

19 days

60

50

40

Experiment A

Wheat 14,8 % Open Barrels

222 days (7 months) 60 50 40 Frequency 30 20 10 0



Fig. 6: Particle moisture distribution measured during storage of farm-fresh and dried wheat in 120-litre-barrels; sampling positions: 1 = circle, 2 = square (see Fig. 3 and Table 2): a) farm-fresh wheat in open barrels (A), b) farm-fresh wheat in closed barrels (B), and c) dried wheat in closed barrels (C). One curve represents the PMD of 150 kernels (50 kernels each from the 3 barrels of one set-up).

4 Conclusions

In this study, the variation of the particle moisture distribution was investigated during laboratory drying and subsequent storage of wheat as well as during pilot-scale storage of storable farm-fresh and dried wheat. For the non-destructive measurement of the single particle (kernel) moisture content, the Time Domain Nuclear Magnetic Resonance (TD-NMR) spectroscopy was applied and adopted. As pretests have shown, the particle moisture significantly varied depending on the angular position in the TD-NMR spectroscope. Therefore, the

kernels were always measured in the same position in the NMR spectroscope, and also the calibration was carried out in this position.

As the basic laboratory drying experiments revealed, the wheat kernels gradually dehydrated as expected and the width of PMD (standard deviation) increased as the drying temperature and the drying time were increased. This was the case even for the best homogeneous drying process attained in the drying chamber with kernels separated on a net. For high drying times at 130 °C this tendency reversed, however. This means, the PMD became narrower associated with a clear over-drying of the kernels. The laboratory storage experiments revealed that the width of the PMD only noticeably decreased when there was enough air available in the container for moisture exchange. During separate storage of single kernels in the micro test tubes, there was no considerable change in the particle moisture distribution. In the glass test tube and, particularly, in the large plastic container, however, the storage process obeyed a clear dynamic of the PMD over time.

The pilot-scale experiments clearly revealed that, if wheat is stored in big storage without ventilation (closed barrels) there is no observable change in the PMD even after months. These experiments were performed so as to simulate dead zones of airflow which can locally occur in real industrial-scale flat stores due to inhomogeneous airflow conditions. The experiments conducted in open barrels showed a drying effect as expected. Generally, the experiments revealed that, both, naturally and technically dried grain have a wider particle moisture distribution than farm-fresh undried grain.

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