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# First shrinkage parameters of Slovak bentonites considered for engineered barriers in the deep geological repository of high-level radioactive waste and spent nuclear fuel

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**Abstract** The high potential of bentonites to volume changes depending on the water content is considered as their advantage for the engineered barriers in the deep geological repository of high-level radioactive waste and spent nuclear fuel because of swelling and self-healing of cracks in contact with water. On the other hand, drying may lead to opening of cracks and spaces between the bentonite blocks. This would increase the permeability and contamination risk around the hot container with high-level radioactive waste and spent nuclear fuel, especially if the host rock mass is dry. First shrinkage tests on four Slovak bentonites studied for engineered barriers were carried out. The water content at the shrinkage limit and the relative linear shrinkage are the first available shrinkage parameters received for the bentonite paste. The shrinkage hazard is higher in the best bentonites with high swelling potential—from Kopernica and Jelšový potok. The results indicated the necessity of further shrinkage tests to determine the relative linear and volume shrinkage of bentonite elements pressed of the loose bentonite powder of low water content.

**Keywords** Radioactive waste · Spent nuclear fuel · Deep geological repository · Slovak bentonites · Shrinkage

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## Introduction

The swelling potential made bentonites to the most recommended material for the engineered barriers in deep geological repositories of high level radioactive waste and spent nuclear fuel, and bentonite emplaced in compacted block form is the preferred option for the clay buffer for most waste management organisations [1–19]. In swelling clays two categories of swelling are observed. The first category—the innercrystalline swelling—is caused by the hydration of the exchangeable cations of the dry clay. The second category—the osmotic swelling—results from the large difference in the ion concentrations close to the clay surfaces and in the pore water. The swelling behaviour of clay rocks depends on the type and quantity of clay minerals encountered their surface charge and the valence of the cations in the double layer [20–25]. In addition, the swelling properties depend on the initial dry density and water content of the soil specimen. The higher the initial dry density, the higher the swelling pressure or the higher the swelling strain; the swelling strain decreases with increasing initial water content, but the swelling pressure seems not to be affected by the initial water content [26, 27]. The swelling properties can be also affected by the chemical composition of the saturating fluid: the swelling capacity of the bentonite decreases with the increase in salinity of saturating fluid, although this influence becomes less significant for higher densities [28–32]. Moreover, studies on the aging effects on the swelling behavior show that the swelling potential may decrease with time due to the rearrangement of clay particles with time [33–35]. The swelling potential of bentonites from several Slovak deposits was already evaluated by the Atterberg liquid limit as indicator, and by swelling pressure tests [36]. While swelling follows wetting, drying leads to shrinkage of bentonites. This seems to be a minor problem for the long-term performance of the barrier expecting that water saturation and swelling will progress fast enough through the clay buffer to limit advective transport. Mayor et al. [37] reported that based on thermo–hydro-mechanical models, bentonite undergoes a drying process due to the sudden increase in temperature close to the canister and reduces the degree of saturation to 0.5 in the first year but then a resaturation begins due to water from the rock mass. Also Pusch et al. [38] point out the necessity of the undisturbed water access for the optimal function of the clay barrier. But gaps between the high-density bentonite blocks used in most repository concepts might open, or fissures in the blocks could appear eventually in the first period of the repository operation, especially if the host rock mass is dry. This could increase the hydraulic conductivity of the buffer and the vulnerability of the host rock mass at least for a short period. To be able to assess the risk of environment contamination by radioactive substances, nuclear fuel fission products included, data on shrinkage parameters of the applied bentonite are necessary. This initiated a pilot research of shrinkage parameters of Slovak bentonites.

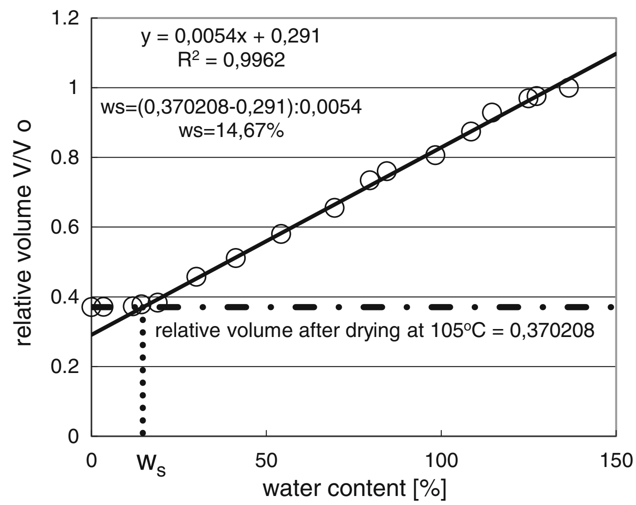


**Fig. 1** Example of the shrinkage limit calculation (specimen LA/1)

## Experimental

### Applied methods

The volumetric shrinkage limit and the relative linear shrinkage were tested. The volumetric shrinkage limit of clays $w_s$ (%) is the water content below which clay ceases to shrink [39]. This limit should be recognized by most of clays by a change to a lighter colour [40]. The research started with a comparison of several technical standards for shrinkage tests which showed that they suggest some more or less different test procedures for $w_s$:

- Method A*: A method for undisturbed or remoulded cylindrical soft rock/soil specimens; their changing water content and related specimen volume are measured during drying and the shrinkage curve necessary for the shrinkage limit approximation is plotted.
- Method A: A volumetric measurement only on the soil fraction passing a specified sieve (mostly 0.5 mm); sieved clay is mixed with distilled water to a smooth homogeneous paste with a moisture content $w$ a little above the Atterberg liquid limit $w_L$, i.e. $w = (1–1.1) \times w_L$. The paste is placed into a shrinkage dish or ring on a glass plate (without entrapping air) and dried at the room temperature. The volume change is monitored during drying and the shrinkage curve plotted as in method A*, while a special attention is given to the colour. At the moment of the colour change, owen drying starts, first at 50 °C, then at 105 °C. Shrinkage limit is estimated from the linear part of the shrinkage curve (Fig. 1).
- Method B: Specimen preparation and drying as in method A, but without continuous measurement, the volume and weight are measured only after full drying. Shrinkage limit is calculated as $w_s = (V_d/m_d - 1/\rho_s) \times \rho_w$, where $V_d$ (cm$^3$) is the volume of the dry specimen, $m_d$ (g) is the dry mass, $\rho_s$ (g cm$^{-3}$) is the particle density and $\rho_w$ (g cm$^{-3}$) is the density of water.
- Method C: No volume measurement; specimen preparation as in method A, drying at the room temperature, monitoring of the specimen weight; here, shrinkage limit is considered as the water content at the moment when the weight lost by drying is lower than 1 % of the previous weight measurement after 24 h.

Table 1 shows types of methods allowed by those national technical standards. There are also different attempts to the shrinkage curve construction within the methods A* and A (different parameters on the *y*-axis), while water content is always on the *x*-axis—read remarks. The practical comparison of the different methods was one of the research aims.

Because bentonite powders were tested (i.e. no undisturbed or compacted samples), the shrinkage limit $w_s$ was measured by the fully monitored method A, that allowed also a calculation according to simplier methods B and C from the measured data and a comparison of results. Specimen dimensions were close to DIN 18122-2 [40] ÖNORM B 4411 [41].

Due to the dimensions of available moulds, the relative linear shrinkage was tested according to BS 1377: Part 2

**Table 1** Shrinkage limit test methods suggested by compared national technical standards

| Technical standard | Suggested method | Remarks |
|---|---|---|
| BS 1377: Part 2: 1990 | A*, A | Specimen dimensions are not specified in the method A*; $d = 44$ mm and $v = 12$ mm in the method A<br>Mercury is used for volume measurement—environmentally no more acceptable<br>Parameter on the *y*-axis of the shrinkage curve: $U = 100 \times V_i/m_d$ where $V_i$ is the volume at the *i*-measurement |
| DIN 18122-2: 2000 | B | Specimen dimensions are $d = 70$ mm, $v = 14$ mm<br>Results are extremely dependent on an exact determination of $\rho_s$ |
| ÖNORM B 4411: 2009 | A, B, C | Specimen dimensions are $d = 70$ mm, $v = 14$ mm<br>Parameter on the *y*-axis of the shrinkage curve: the relative volume $V_i/V_o$ where $V_o$ is the volume of the shrinkage dish/ring, i.e. the initial clay volume |
| STN 72 1019: 1989 | A* | Parameter on the *y*-axis of the shrinkage curve: the relative volume shrinkage $v_s = (V_i - V_d)/V_d$<br>Specimen dimensions are $d = 60$ mm, $v = 45$ mm<br>The same specimen is used for the linear shrinkage and for the shrinkage limit determination<br>A special dimension measuring apparatus is necessary<br>Drying must be slow, the drying rate should be controlled by a container to avoid fissures |

[39] and ÖNORM B 4411 [41]. Sample preparation was the same as in the shrinkage limit test. The relative linear shrinkage $L_s$ was calculated as $L_s = (1 - L_d/L_o) \times 100$, where $L_o$ is the initial length of the sample and $L_d$ is the length of the dry sample.

Bentonite samples

Four bentonites from deposits in Slovakia were tested: J250 from Jelšový potok, K45 from Kopernica, L45 from Lieskovec and LA45 from Lastovce. Many authors give their exact geological, mineralogical and geochemical characteristics, the most recent review of those publications was set up by Šucha, Andrejkovičová, Stríček, Osacký, Melichová and Galamboš et al. [42–54]. The number in the bentonite symbol gives the grain size, e.g. J250 means grains passing the sieve mesh 0.250 mm. Data on Atterberg consistency limits and particle density were taken from publication [55].

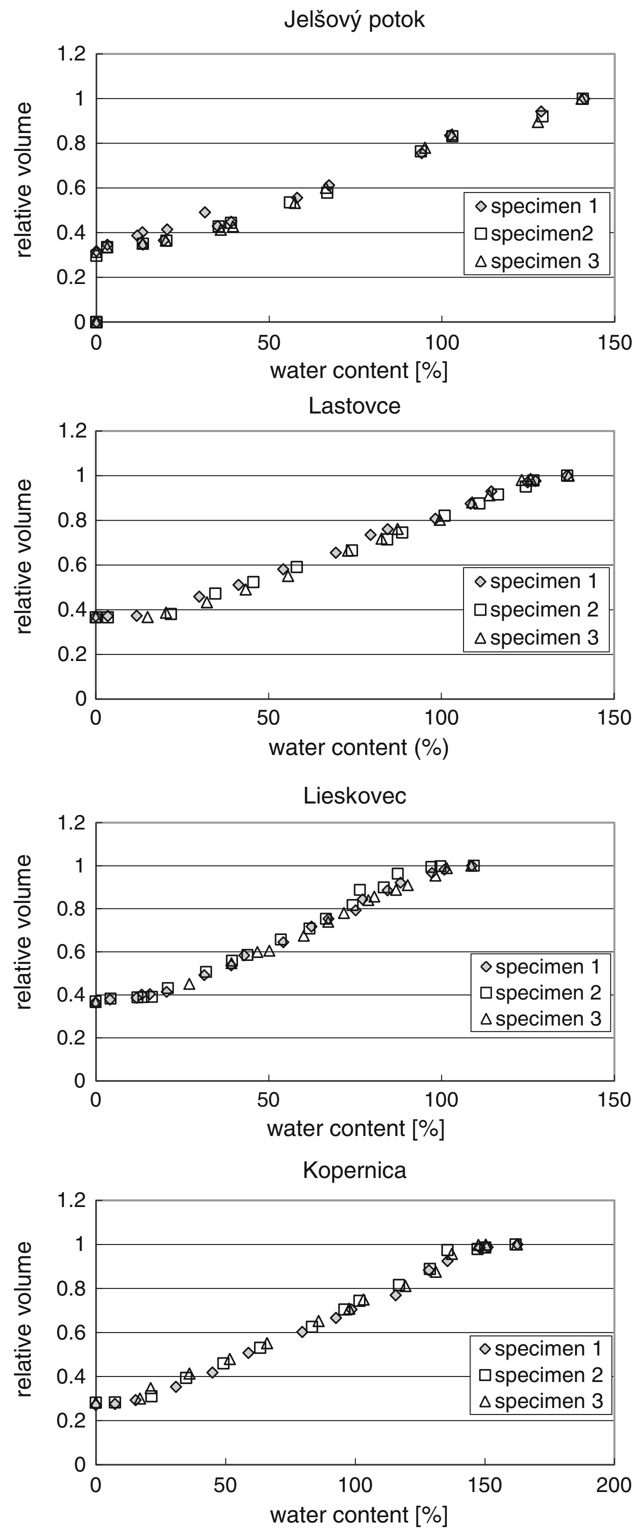


**Fig. 2** Results of the shrinkage limit tests (volumetric method A)

## Results and discussion

The results of the shrinkage tests are given in Fig. 2 and Table 2. If the shrinkage limit $w_s$ is lower than the equilibrium vapour content at room conditions $w$, bentonite blocks might shrink and gaps between them open due to high temperature around the radwaste containers until water from the host rock mass intrudes and bentonite swells. This could happen to the blocks of bentonites J250 and K45 according to test methods A and C, while no hazard was indicated by the method B. Bentonites L45 and LA45 show no shrinkage hazard. Because of different results by different methods, further shrinkage tests are necessary. Shrinkage of the compacted high-density bentonite should be studied, because shrinkage limit of all studied bentonites is very similar, but the relative linear shrinkage $L_s$ shows differences. Bentonites K45 and J250 showed the highest relative linear shrinkage, as these bentonites have also the highest swelling potential, i.e. the potential to volume changes [55].

The experience with the different tests methods was summed up as follows. The determination of the relative linear shrinkage $L_s$ showed very high values, but it does not indicate the ultimate shrinkage of compacted bentonite blocks. This test is suitable explicitly for comparative studies when searching for material less sensitive to shrinkage. In tests on compacted bentonites (as in method A*), shrinkage depends not only on the current water content, which is far below the liquid limit required as starting moisture in standard tests, but also on the grain size distribution and porosity (pore number and size).

It is to point out that different interpretation (methods A, B and C) of the same shrinkage limit tests with the same specimens brought different results. One should be aware of this and always ask about the exact description applied for $w_s$ determination. The application of the method B is questionable, because results are highly dependent on the accuracy of the problematic particle density determination. Small deviation makes big difference in the shrinkage limit.

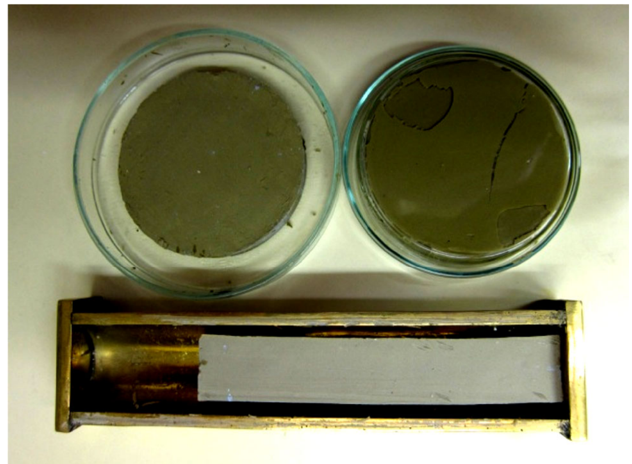

**Fig. 3** An example of shrinkage tests. *Upper* shrinkage limit test: specimen colour at the shrinkage limit (*left*), colour etalon of a liquid paste for comparison (*right*). *Below* determination of the percentage of the linear shrinkage in a standard mould, colour of the same bentonite after drying at 105 °C

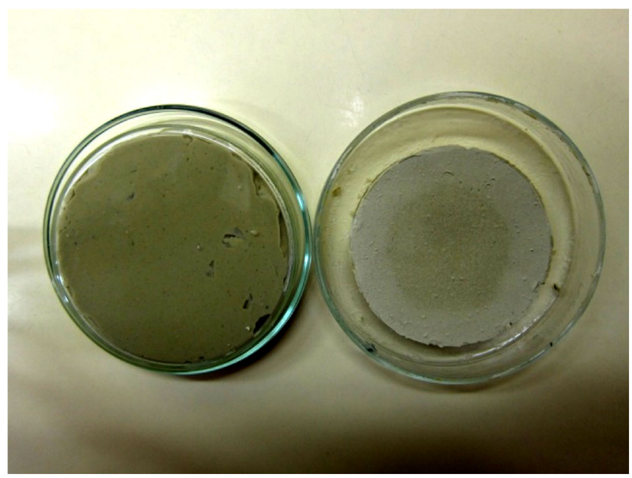

**Fig. 4** Colour change could be well observed in the bentonite L45. Specimen centre dried as last, creating a darker part with a lighter rim of the dry bentonite

**Table 2** Test results

| Bentonites | $w_s$ (%) Determination methods A | B | C | $L_s$ (%) | $w^a$ (%) | Colour of the wet etalon | Colour of the sample dried at 105 °C |
|---|---|---|---|---|---|---|---|
| J250 | 13.26 | 15.06 | <13.45 | 32.26 | 14 | 5Y 5/6 light olive brown | 5Y 8/1 yellowish grey |
| K45 | 13.75 | 15.28 | 14.87 | 33.26 | 15 | 5Y 5.5/1 gray | N 6.5/0 light grey |
| L45 | 13.07 | 15.71 | <12.0 | 26.42 | 10 | 5Y 5/3 olive | 5Y 6/4 pale olive |
| LA45 | 13.75 | 15.28 | 14.87 | 26.95 | 11 | 5Y 6/2 light olive grey | 2,5Y 8/1 white |

[a] Adsorbed vapour/water content in the untreated bentonite in the equilibrium with the air humidity at room conditions

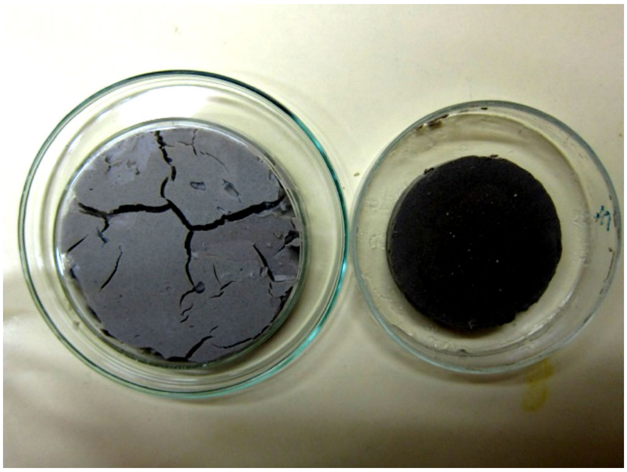

**Fig. 5** Bentonite K45 at the shrinkage limit (*right*) was still darker than the colour etalon (*left*)

The colour change observation does not work well as indicator of reaching the shrinkage limit. Some bentonites (J250, L45—Figs. 3, 4) became lighter than the liquid paste (method A), but the daily mass decrease by drying was still above the 1 % defined as indicator of the shrinkage limit in the method C. On the other hand, slowly dried K45 was still darker than the fresh paste, even after drying at 50 °C (Fig. 5). Finally, its colour got lighter only after drying at 105 °C.

Some bentonites (e.g. J250) cracked during free drying. But if the drying rate is controlled by containers as in STN 72 1019 [56], method C cannot be used.

## Conclusion

A comparison of Slovak, Austrian, German and British Standards for shrinkage test methods showed that Slovak Technical Standards (STN) pertain to undisturbed soil samples, where shrinkage depends not only on the current moisture, but also on the grain size distribution and porosity. Shrinkage limit $w_s$ (%) and relative linear shrinkage $L_s$ (%) determined according to the foreign technical standards are independent from those properties, because sample preparation brings all soils to equal starting conditions: sieved to grain size below 0.5 mm, water added to reach moisture $w$ near the liquid limit $w_L$, suspension smeared without bubbles into standard moulds. Applied Austrian ONORM B 4411 2009 offers three test procedures for $w_s$. First, $w_s$ was determined by manifold measuring of the sample weight and volume during drying. Calculation followed from the water content versus relative volume plot with a linear trend line. Results were compared to data from the other two methods. The highest $L_s$ (up to 33.3 % of the initial length) was observed in the bentonite K45 from Kopernica and in J250 from the Jelšový potok deposit (32.3 %), the best Slovak bentonites because of their swelling potential and excellent adsorption properties for radionuclides. Reaching of the shrinkage limit ($w_s = 13$ %) of J250 was indicated also by a colour change from light brownish grey (2,5Y 6/2) to light grey (2,5Y 7/1—Munsell Soil-Color Charts, 2009). Because $w_s$ obtained by two test methods is lower than the equilibrium moisture of the bentonite J250 under room conditions, which is 14 %, bentonite blocks might shrink and gaps between them open due to high temperature around the containers with high-level radioactive waste and spent nuclear fuel in the deep geological repository, until water from the host rock mass intrudes and bentonite swells. The same conclusion about the shrinkage hazard was drawn for the bentonite K45 from Kopernica. Results indicated the necessity of further shrinkage tests, but on bentonite powder compacted to high-density blocks/segments. Methods described in STN 72 1019 [56] are recommended as relevant for the assessment of their total linear and volumetric shrinkage.

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