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Coupling between leaching and creep of concrete

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Abstract

In a radioactive waste disposal, concrete containment structures must be studied over extended periods during which it is necessary to account for a possible degradation by calcium leaching due to on-site water. An experimental investigation is described where the effects of an accelerated calcium leaching process of concrete on creep of concrete are highlighted. The comparison with a creep test where the sample is immersed in water shows that leaching generates tertiary creep and rupture of the specimen. A Dirichlet series coupled to a mechanical damage are used to model the coupled tertiary creep. With this method we can evaluate the lifetime of concrete structures subjected to chemical and mechanical loading.
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Keywords: Concrete; Leaching; Creep

1. Introduction

Radioactive waste storage structures must be studied over periods one order of magnitude or more times greater than those of conventional civil engineering structures. This means that degradation phenomena normally only considered very rarely have to be taken into account. Leaching of calcium contained in concretes by water from the surrounding rock is one such case. This phenomenon leads to considerable change in the microstructure taking the form of increased porosity [1] and degradation of mechanical properties [2–6]. Leaching is also closely coupled with mechanics [3–5, 7–10] and influences the delayed behavior of concrete [11,12]. In the first section we will show that if leaching occurs under a sustained load, creep leads to the ruin of the material. We will then propose an analysis aiming to evaluate the lifetime of structures submitted to creep and leaching.

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2. Experimental results

2.1. Concrete formulation

The concrete studied is defined in Table 1. Its aggregates are siliceous and therefore resistant to leaching. Our samples are cylinders 11 cm in diameter and 33 cm high. These samples were removed from their mould the day after their manufacture and kept in water for 9 months. They were then ground to obtain good surface flatness before loading.

2.2. Leaching

Natural leaching is a very slow process (a few centimeters per hundred years). For laboratory experiments, the use of deionised water is not an optimum choice for concrete for which we need several centimeters of degradation (to have a representative volume compared to the size of the aggregates for instance). Accelerated leaching is necessary to leach the samples in a reasonable time. There are three principal ways to

Table 1
Formulation of the studied concrete

Components	Quantity (kg/m^3)
Siliceous sand (0,2–3,15 mm)	684
siliceous gravel (4–12,5 mm)	1050
Cement CEMI	375
Water	225

accelerate calcium leaching: using temperature [13], using an electrical field [3,37] and by replacing deionized water with another solution agent to increase concentration gradients between the interstitial solution and the aggressive environment. The majority of the experiments on calcium leaching of cementitious material samples are performed by using the last method. The deionized water is replaced by a strongly concentrated ammonium nitrate solution [14,2,15,3,16,17, 6,13]. Here, we use an ammonium nitrate solution at a concentration of 6 mol/l. It has been shown that the attack of this solution is close to that of deionised water, but with kinetics roughly 200 times faster [2,3,16,4,13].

Leaching is accompanied over time by the propagation of the degradation front corresponding to the dissolving of portlandite. The pH value in the pore solution of cementitious materials is higher than 12.5, creating a very alkaline environment. Consequently, an ammonium nitrate solution with pH values below this level characterizes an acid environment.

The degraded depth is determined by using the phenolphthalein on sectioned samples. We use a pH indicator like phenolphthalein to distinguish between the sound zone and the degraded zone. Phenolphthalein turns from colorless in acidic solutions to pink in basic solutions with the transition occurring around pH 9. But the dissolution of portlandite occurs as long as the pH values drop below 12. Therefore, phenolphthalein does not give the exact position of the dissolution front of portlandite. However, by comparison between the measurement by phenolphthalein and by SIMS microprobe analysis, it was shown by Le Bellégo et al. [17] that for the cement paste the total degraded depth e_t can be determined by correcting the degraded depth e_{phenol} measured by phenolphthalein.

Monitoring of this front over time shows evolution in proportion to the square root of time until it reaches the center of the sample (Fig. 1). This result is characteristic of diffusive phenomena and has been observed by many authors [1–3,16,4,18] and corroborated by theoretical considerations [19].

2.3. Leaching under load

2.3.1. Description of tests

Two creep test frames were used. Each has a particular function:

- one frame is used for a control basic creep test in water,
- the other one is used for the creep test with leaching by ammonium nitrate at 6 mol/l and allows combined chemical and mechanical effects to be observed.

Two similar samples, made from the same batch, were tested: one in water and the other one in ammonium nitrate solution.

An experiment setup was specially designed for these tests. Firstly the test pieces were surrounded by plastic cylindrical recipients containing liquids (water or the ammonium nitrate solution). Impermeable seals were fitted to prevent any leakage. Secondly, the parts outside the recipients were covered with a silicon resin and plastic film to prevent extraneous diffusion into the test pieces or drying. Fig. 2 shows the ammonium nitrate test bench.

The test principle consists in compressing the concrete test piece between the plates of the creep frame at a constant force over a long duration.

The force applied is kept constant using a nitrogen–oil oleopneumatic accumulator (see Fig. 2). The load is applied by a flat jack fed with oil by a hand pump. The force aimed for is 25% of the resistance of the test piece, i.e. roughly 10 MPa. First the oleopneumatic accumulators are inflated using a nitrogen cylinder at a pressure equal to 80% of the required oil pressure. The test pieces are then installed on the creep test frames, and the recipient is filled with ammonium nitrate. Another large recipient is used to renew the ammonium nitrate (see Fig. 2) so that the pH remains sufficient for leaching. Whenever the pH reaches 8.2, the solution is renewed. An electrode and thermal probe are installed to monitor the pH and temperature of the solution. Movement between the plates is measured by an LVDT movement sensor. All the systems are connected to a computer for data acquisition. Finally, the load is applied to the test piece by increasing the flat jack pressure with the hand pump. The oil pressure is monitored by a pressure sensor. The oil in the oleopneumatic tank is then connected to the oil in the flat jack and the nitrogen tank compensates to a large extent the effect on the oil pressure caused by deformation.

The force applied to the test piece is verified by lifting off the upper load cell. This is done at set times to check the constancy of the force applied. If there are losses, oil is added to

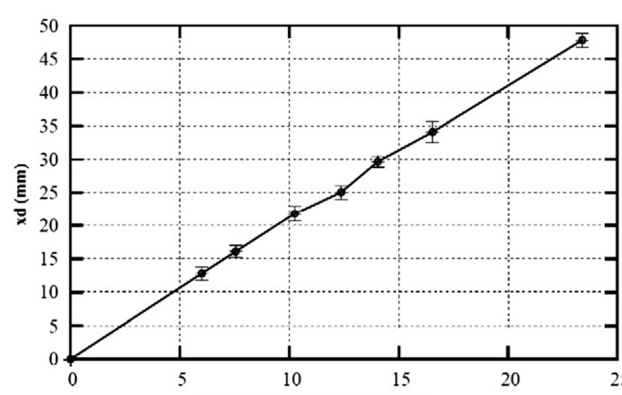

Fig. 1. Evolution of degraded depth over time: $x_d = k\ t^{0.5}$ where x_d is the degraded depth expressed in mm and t is time in days. This relation was established through testing without mechanical load [9]. The error bar represents minimum and maximum values of the degraded depth taken on the same sample at the same time.

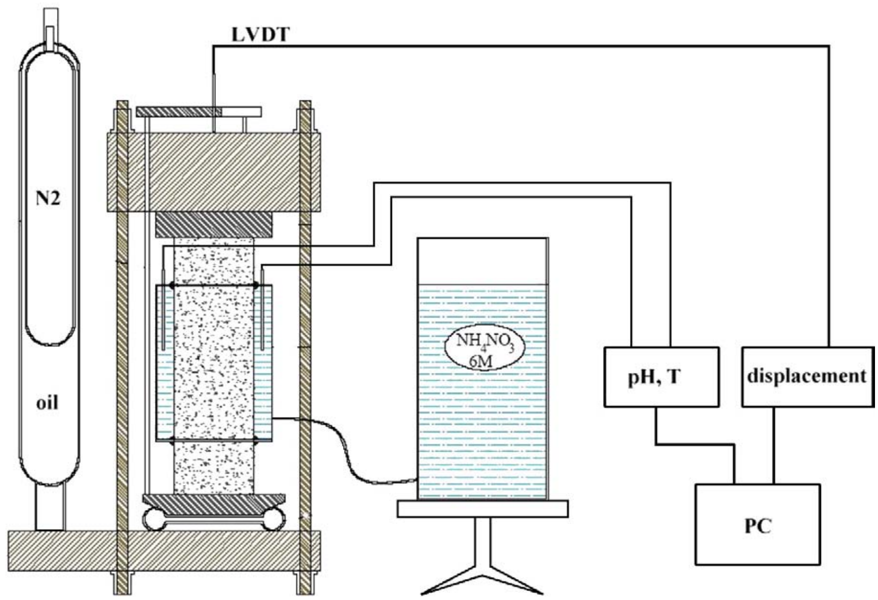

Fig. 2. Diagram of the test bench for creep combined with leaching.

compensate for them. In our test, the lift-off force F is 105 kN, corresponding to mean stress of 10.5 MPa.

2.3.2. Test results

Fig. 3 shows the evolution of deformation for both tests (the experimental results could be downloaded from http://perso.lcpc.fr/jean-michel.torrenti/). Initially, considering the scattering of the measurements (about $\pm 100\ 10^{-6}$), there is very little difference between the two tests. Then deformation in the combined test accelerates and there is a difference larger than the scattering. Firstly, as it will be shown later, this acceleration corresponds linearly to the increase of stress (linear creep). Then a tertiary creep phase is reached and leads to ruin. This demonstrates the effect of creep combined with leaching. Similar results have been obtained with concrete containing calcareous aggregates by Lacarriere et al. [12].

The test was interrupted after 435 days and a cross-section of the leached test piece was made. The leached area could then be revealed using the colored indicator phenolphtalein. As said before, this indicator does not reveal the exact position of the degradation front and a correction factor must be applied [17]. The mean degraded depth determined by experiment is therefore 29 mm.

Until the degraded depth reaches the center of the sample (this is not the case in our test because mechanical failure occurs before), the mean degraded depth could be expressed by:

$$x_{\mathrm{d}} = kt^{0.5} \tag{1}$$

The experimental value corresponds to $k=1{,}4$ mm $\mathrm{d}^{-0{,}5}$ and can be compared with the degraded front one obtains without mechanical loading for $t=435$ days: 44 mm (see Fig. 1). We have a smaller degraded depth that can be explained by two ways:

- there is a coupling between diffusivity and the stress state of the leached material. Nicolosi [10] has shown that a coupling between the chemical process and the mechanical behavior is possible through the effect of the interstitial pressure. But his results indicate a small influence. We can also think to a small influence of the mechanical loading on the porosity;
- in spite of the pH control, the boundary conditions for leaching in the creep test are not exactly the same compared to the leaching test without mechanical load. A lower renewal of the solution around the sample may explain a lower degradation.

We think this second possibility is the more likely because the degraded depth was variable around the sample indicating perturbed boundary conditions but another test would certainly be useful.

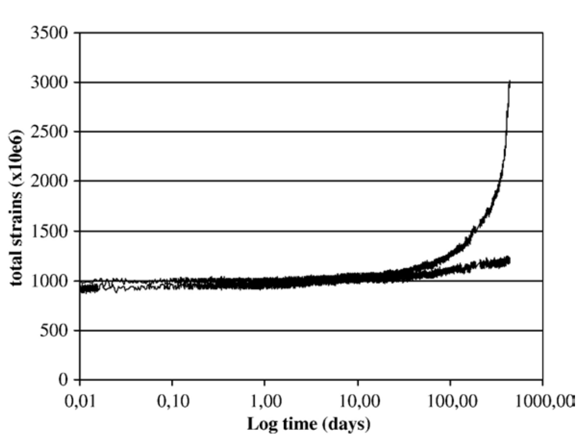

Fig. 3. Total strains under water and in the ammonium nitrate solution.

3. Modeling

3.1. Modeling basic creep—Dirichlet series development

As proposed by Bazant and Chern [35] we use a description in the form of a Dirichlet series (which correspond to Kelvin–Voigt elements placed in serial) to model the concrete creep:

$$\varepsilon_{zz,fl}(t) = \sum_{s=1}^{n} \int_{\tau=0}^{t} Js[1-\exp(-t/\tau s)]\,\dot{\sigma}_{zz}(\tau) \quad d\tau \tag{2}$$

where J_s stands for the compliance and τ_s for the characteristic time of one component of the series and $\sigma_{zz}(t)$ for the stress rate due to the decrease of the sound part of concrete. Breaking down the creep strain $\varepsilon_{zz,\mathrm{fl}}$ on the basis of $\{1, \exp(-t/\tau_i)_{i=1,n}\}$ produces [36]:

$$\varepsilon_{zz,\mathrm{fl}}(t_i) = A_0^i + \sum\nolimits_{s=1}^{n} A_s^i \tag{3}$$

Knowing the creep strain $\varepsilon_{zz,\mathrm{fl}}(t_i)$ at time t_i makes it possible through recurrence to construct the response to time t_{i+1}, $\varepsilon_{zz,\mathrm{fl}}(t_{i+1})$:

$$A_0^{i+1} = A_0^i + \Delta\sigma_{zz}(t_{i+1}) \quad \sum\nolimits_{s=1}^{n} Js(t_{i+1}) \tag{4}$$

$$A_s^{i+1} = A_s^i \quad \exp\left(-\frac{t_{i+1}-t_i}{\tau s}\right) - \Delta\sigma_{zz}(t_{i+1}) Js(t_{i+1}) \exp\left(-\frac{t_{i+1}-t_i}{\tau s}\right) \tag{5}$$

All that is required therefore is to store the $n+1$ A_j at each time step. The functions $J_s(t_i)$ should enable the various effects to be taken into account, particularly the ageing of concrete or, in the problem concerning us, the effect of leaching. The effect of ageing will be neglected in the following. Fig. 4 presents the adjustment to the Dirichlet series for the creep itself (sample in water). The parameters are adjusted in order to be positive and to follow the strain considering the scattering.

It should be noted that such a model is incapable of recovering irreversible basic creep strains at unloading, which account for about 60 to 70% of the total creep strain. This is not a critical issue if only constant or increasing stress evolutions are involved (which is the case here). In order to analyze the results of the leaching creep test, we can assume that the stress taken up by the degraded area could be neglected. This is a realistic hypothesis as the degraded area can only take up a limited fraction of the initial resistance [2,3,5,9].

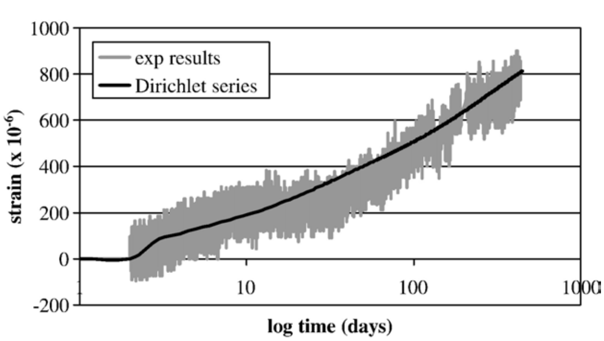

Fig. 4. Adjustment of the Dirichlet series to the basic creep of concrete—$n=7$; $t1=0{,}002$ days; $t2=0{,}02$ days; $t3=0{,}2$ days; $t4=2$ days; $t5=20$ days; $t6=200$ days; $t7=2000$ days; $J_0=1/36.2$ GPa^{-1}; $J_1=1/800$ GPa^{-1}; $J_2=1/800$ GPa^{-1}; $J_3=1/400$ GPa^{-1}; $J_4=1/200$ GPa^{-1}; $J_5=1/50$ GPa^{-1}; $J_6=1/25$ GPa^{-1}; $J_7=1/17$ GPa^{-1}.

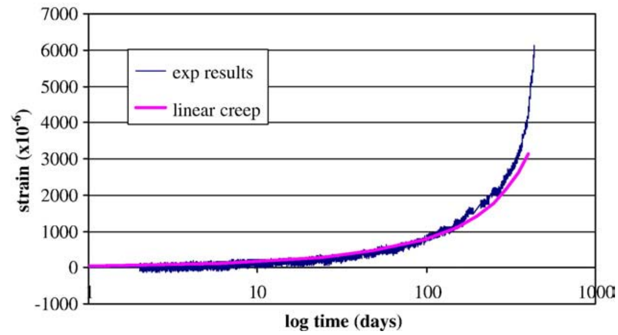

Fig. 5. Comparison between the delayed strain in the leached test and a linear evolution of the strain due to evolution of the stress.

Assuming the degraded depth is the experimental mean degraded depth ($k=1{,}4$ mm $d^{-0{,}5}$); it is possible to calculate at each time the stress $\sigma(t)$ which is applied to the sound part of the concrete:

$$\sigma_{zz}(t) = \sigma_{zz}(t_0)\frac{\pi R^2}{\pi\,(R-k\sqrt{t})^2} \tag{6}$$

From this, the evolution of the creep strain is evaluated assuming linear creep. At the beginning of the test, the delayed strain could be well explained with a linear creep (Fig. 5). But, as the stress increases a difference appears between experimental strain at the end of the test and evaluation using linear creep. This difference ($3000\ 10^{-6}$) is larger than the scattering ($200\ 10^{-6}$). In fact, the creep strain becomes non linear and we have to take into account tertiary creep.

When a concrete specimen is subjected to a creep test, using acoustic emission Rossi et al. have shown that a microcracking occurs [20]. If the stress applied exceeds approximately 80% of the material strength in compression, since Rüsch et al. we know that ultimately the material ruptures [21]. This phenomenon is called tertiary creep and is related to a strong interaction between the creep and the damage of the materials [22–27].

This coupling can be taken into account by means of a visco-elasto-plastic model [28] or by a coupling between creep and damage [12,29,30]. Here, following an idea suggested by various authors but applied to Maxwell chain models [26,27,31] the coupling with the damage is taken into account by assuming that all the terms of the Dirichlet series are affected by a scalar damage variable $d(t)$.

This is equivalent to introduce an effective stress such as:

$$\sigma = (1-d)\cdot\tilde{\sigma} \tag{7}$$

Relationships (4) and (5) are maintained with:

$$J_0(t_i) = J_0/(1-d(t_i)) \tag{8}$$

and

$$J_s(t_i) = J_s/(1-d(t_i)) \tag{9}$$

and Eq. (2) is generalized in 3-D by introducing a creep Poisson ratio such that:

$$\varepsilon_{rr,\mathrm{fl}} = \varepsilon_{\theta\theta,\mathrm{fl}} = -\nu_{\mathrm{fl}}\varepsilon_{zz,\mathrm{fl}} \tag{10}$$

Changes in the damage are as proposed by Mazars [32]. The threshold function is as follows:

$$f(\tilde{\varepsilon}, d) = \tilde{\varepsilon} - \mathrm{K}(d) \tag{11}$$

with K a damage parameter, equal to the maximum value reached by the equivalent strain $\tilde{\varepsilon}$ during the loading history, with an initial value equal to K_0. The equivalent strain is defined by:

$$\tilde{\varepsilon} = \sqrt{\sum \langle \varepsilon_{\mathrm{ii}} \rangle_+^2} \tag{12}$$

where $\langle \varepsilon_{\mathrm{ii}} \rangle_+ = 0$ if $\varepsilon_{\mathrm{ii}} < 0$ and $\langle \varepsilon_{\mathrm{ii}} \rangle_+ = \varepsilon_{\mathrm{ii}}$ if $\varepsilon_{\mathrm{ii}} > 0$. Following an idea suggested by Omar et al. [27], Mazotti and Savoia [26] and Reviron et al. [33], we define $\varepsilon_{\mathrm{ii}}$ as being the sum of the instantaneous extensions and a fraction of the delayed extensions:

$$\varepsilon_{\mathrm{ii}} = \varepsilon_{\mathrm{ii\_inst}} + \beta\varepsilon_{\mathrm{ii\_fl}} \tag{13}$$

β being an adjustment coefficient. Fig. 6 presents the creep deformation simulations obtained with the model adjusted to Fig. 4 coupled with the damage, the additional hypotheses being: the creep Poisson coefficient ν_{fl} is assumed equal to 0.2, value of the instantaneous Poisson coefficient and β=0.05. The obtained value of β is less important than the one obtained by Mazotti and Savoia [26] (β=0.1−0.2) and Reviron et al. [33] (β=0.24). In the first case, the authors use a different damage model and in the second case it was assumed that damage affects only the elastic part. Concerning the creep Poisson ratio, experimental results show a increase in the tertiary phase, but this has not an impact on the prediction of uniaxial strains or the lifetime.

Finally, knowing the creep strains and the evolution of the compliance, we can calculate the total delayed strain $\varepsilon_{\mathrm{del}}$ which

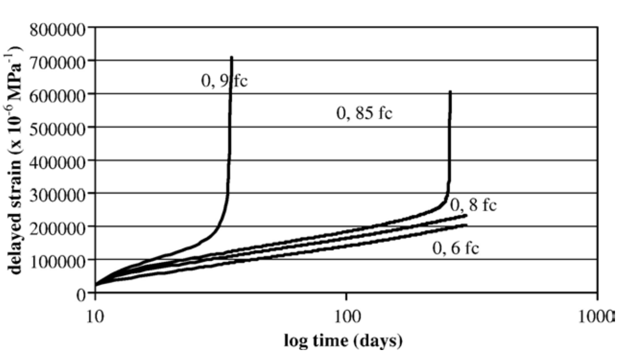

Fig. 6. Evolution of the delayed strains divided by the applied stress when this stress is varying from 0.6 fc to 0.9 fc. The parameters of the Dirichlet series are those of Fig. 4. For the damage model Ac=1.5, B_c=1500, K_0=120 10^{-6} (Mazars [32]).

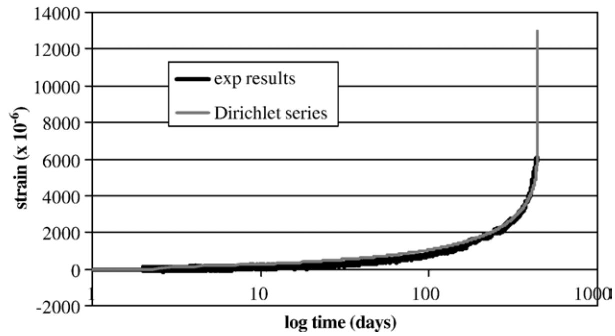

Fig. 7. Creep test under leaching—comparison between experimental results and modeling. The parameters of the Dirichlet series are those of Fig. 4 and the parameters of the damage model are those of Fig. 6.

includes also the variable part of the instantaneous strain like in the experimental measurement:

$$\varepsilon_{zz,\mathrm{del}}(t) = \varepsilon_{zz,\mathrm{fl}}(t) + J_0(t)\sigma_{zz}(t) - J_0(t = t_0)\sigma_{zz}(t = t_0) \tag{14}$$

where t_0 is the time when loading is applied. It is this strain $\varepsilon_{zz,\mathrm{del}}(t)$that will be compared to experimental results.

3.2. Creep coupled with leaching

For the instantaneous part of the model we have the classical relation:

$$\sigma_{ij} = (1 - d)\Lambda_{ijkl}\varepsilon_{kl} \tag{15}$$

$$\text{with}(1 - d) = (1 - d_{\mathrm{ch}})(1 - d_{\mathrm{m}}) \tag{16}$$

as proposed by Gérard [3] and Carde [34] where d_{ch} is the chemical damage and d_{m} is the mechanical damage.

In our case, the stress taken up by the degraded area is neglected. This is equivalent to take $d_{\mathrm{ch}} = 1$ in the degraded area. In this case, J_s are only affected by the mechanical damage.

At each time step the stress $\sigma(t_i)$ which is applied to the sound part of the concrete is given by Eq. (1).

Using the model developed in Section 2.2, it is possible to calculate the delayed strains of the specimen which is leached (Eq. (14)). The calculation is halted when damage d reaches 1. Fig. 7 shows the result of the modeling compared to the experiment. The estimation of the service life is correct. We therefore have a simplified method of forecasting compressive loaded structures subject to leaching.

4. Conclusions

Experimental results show that there is a coupling between creep and leaching. The deformation in the combined test accelerates to reach a tertiary creep phase leading to the ruin of the concrete.

Using Dirichlet series for creep coupled with a scalar damage, we are able to reproduce this phenomenon and to predict the service life of the sample subjected to a sustained compressive loading and to leaching.

Of course we have used in our test an accelerated method and the in-situ kinetics of the phenomenon would be much slower (about 200 times). Ageing of creep would slow the delayed strains and consequently the mechanical damage. But it seems very important to account for the coupling in the study of the long-term behaviour of the storage and evaluate its lifetime.

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