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CONSERVATION OF ARCHAEOLOGICAL MEGALITHIC SITES UNDER MARINE ENVIRONMENT: EXAMPLE OF THE GRANITIC MENHIRS AT I STANTARI (CAURIA PLATEAU, CORSICA ISLAND, FRANCE)

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Abstract

Granitic menhirs from the Neolithic to Bronze Age period are present in few places of Corsica Island (France) such as I Stantari alignments on Cauria plateau. The emblematic alignment of statue-menhirs of Stantari shows various degradation features. They are characteristic of granite stones alteration and lead to the disappearance of the fine sculpted relief. A first campaign of conservation work using acrylic resins as consolidant was performed in situ in 1993. Ten years after this intervention, the archaeologists who were working on the site, asked for an evaluation of the efficiency of the treatment. The performance of this impregnation of the whole monoliths has been evaluated by means of visual examination, ultrasonic velocity measurements and microanalyses performed on several samples in the laboratory. The investigations have shown that the intervention didn't succeed to improve the conservation of the sculptures. The properties of the selected consolidant product, the application mode and the deficient diagnosis were involved in this failure. An experimental procedure was then set up to test new products that could be applied on the granitic stones. Two acrylic resins including the one yet used, ethyl silicate and ethyl silicate combined with a silane resin were tested as strengthener. None of these products are efficient enough. The causes of the degradation and the adequacy of the product application are discussed in regard to the specific properties of the stone material.

Keywords: megaliths, granite, degradation patterns, ultrasonic velocities, consolidants, experimental ageing.

1. Introduction

I Stantari menhirs and statue-menhirs alignment is one of the main megalithic sites of Corsica Island (France). They are located near Sartène on Cauria plateau at a few kilometres from the seaside (Figure 1) and were discovered during archaeological excavations (Figure 2). Only 7 were still visible during the XIXe century as Adrien de Montillet described it. Robert Grosjean's team dug out others in 1964 and 1968 (In d'Anna *et al*, 2004). In 1975, some menhirs belonging to the alignment were set upright again in order to re-establish the alignment of 12 raised monoliths that are currently visible. Their orientation is north-south. They were probably initially dressed up at the beginning of the final Bronze Age, towards 1000BC.

Deterioration issues were first described after Grosjean's excavation campaign in 1968. The degradation partially erased the relieves on several menhir-statues. A first study was made in 1993 in order to establish a degradation diagnosis and to propose a treatment protocol to stop the degradation (Casta, 1993). A strengthening treatment was then applied in situ on most of the raised megaliths (Casta and Poli, undated).

During the 2002-2004 periods, d'Anna et al. (2004) who was starting new archaeological excavations on the site considered that a new diagnosis was required. They were wondering what had been the effects of the treatment. They asked for a new diagnosis of the granite degradation including a condition report of the raised megaliths.

The current study deals with the evaluation of the 1993 intervention, the diagnosis of the stone degradation, and laboratory testing of new products that could be used to consolidate the granite.

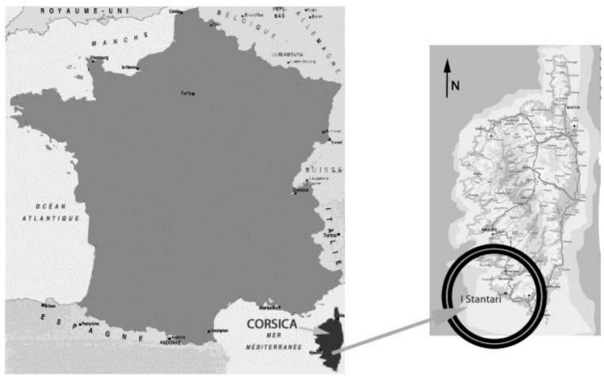

Figure 1. Location of the site of I Stantari (Corsica Island, France).

Figure 2. View of I Stantari's site during the excavation campaign in 2007 and its erected and strengthened monoliths (11 are visible and are referenced in the insert).

2. Description of the raised megaliths and their state of conservation

Megalithic granite is a monzogranodiorite. Microgranites and aplitic sills locally cross it. It is constituted in quartz, feldspars, biotite and amphiboles. The potassic and calco- sodic feldspars are about 1 to 2 cm in size (J.-B. Orsini in d'Anna et al., 2004).

Granite megaliths of I Stantari's site show degradation patterns. Several networks of fissures affect them: the largest ones mark the stone foliation and interganular microfissures lead to the granular disintegration of the stones. The cracks network is very important. The main ones are vertical and parallel to the granite foliation. Flashings used in 1993 plug them. The other are inter or intra minerals and mainly affect feldspars.

The statue-menhirs present faces and several armed warriors facing east. The surface of the stone is eroded. The carved faces, swords etc. are difficult to see. The exposed West face of the erected stones to the prevailing wind and the rain is rougher than the opposite one. Granular disintegration affects also the West faces of the megaliths. East faces and mostly their bottom show desquamations and blistering. Scaling and locally blistering are visible on some monoliths (M2 and M4). The scales are few millimeters thick and bases of blistering are 4 to 5 millimeters in diameter. M5 monolith shows little white craters few millimetres to one centimetre in diameter. These degradations locally lead to a loss of materials.

A red colouration locally affects the erected stones except in their excavated parts. This reddening is probably due to fires.

Past and recent biological colonisations (mostly mosses and lichens) are locally present on the monoliths, mainly in their upper part. Lichens are grey or have a green to yellow colour. Some are under the consolidant film, other developed themselves on it.

All of these alterations affect also the granitic outcrops, which would be the quarry.

3. Evaluation of the 1993 intervention

According to Casta (1993) and Casta and Poli (undated), biological colonisations described as lichens were cleaned using hydrogen peroxide. Scales were stuck using an Acryl™ AC261K (Röhm and Haas) associated to a matting agent and the large fissures were filled by application of a flashing with the same resin. The strengthening impregnation using this water dispersed acrylic resin had been performed by infiltration from the upper part of the stones in two steps, first 40% in dilution, then at 60%. This method was preferred to brush application to have the best penetration of the resin (Casta and Poli, undated). It was expected to give a homogeneous repartition of the resin; a long contact with the stone which should allowed making easier the absorption.

The observations in 2006 and 2011 showed that the degradation is still active but it seems to be slow. Because of the age of these cultural heritage objects and the shallow residual engravings, the degradation has to be stopped and a precise conservation policy has to be carrying out (Vallet et al., 2008). First, Casta's consolidant has been characterised using spectrometries Raman and FTIR and the nature of the product was able to be determined as a poly(nButyle Acrylate- MethylMethacrylate) which is equivalent to the current one (Rogalle, 2006). FTIR spectra revealed also the presence of polyethylene glycol, which shows a degradation of the resin.

The surfaces aspect did not change a lot. No new fissure and no new coloration appeared. Lichens size was quite constant. Some monoliths (M2, M4, M5, M7 and M12) show new degradation patterns mostly on their oriented East surface. Chatain (2010) underlined that these monoliths and their East surface were already the most sensitive to the degradation. Scaling and blistering were in progress, mainly on the lower part of the East oriented faces. One can see the discontinuous film on the surface (Figure 3). There are locally lichens, green algae under it. The surfaces of the monoliths seem to be washed. Their base and the upper part of the surrounding soil have been strongly hardened. In 2008, the surface was so fragile that it was impossible to make small cores.

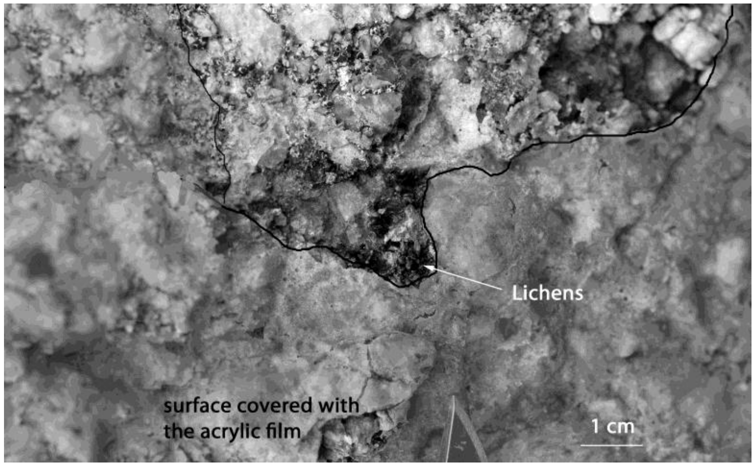

Figure 3. Detail of the surface showing that the acrylic film does not cover the entire surface and that there are some lichens which grow.

Ultrasonic velocities measurements were performed in 2005 and 2011 to study the cohesion of the 12 monoliths and to evaluate the residual consolidant effects. An AU2000 from CEBTP (60 kHz) which measures the times that the ultrasounds take to cross the stone has been used in transmission. They were performed from bottom to top on the West-East faces and also along the North- South direction on the same spotting points. 3 measures have been done on each point.

Megaliths show sound velocities less than 3000m/sec. These velocities (megaliths 2 and 7 excluded) are equivalent to those measured on walls from the nearest granitic outcrops (from 1962 to 2729 m/sec; see Figure 4). Compared to the velocities value on fresh granite (6000 m/sec), these ultrasonic velocities are very low.

Some variations occurred from 2005 to 2011 campaigns (Figure 4). Most of the megaliths show the same average velocity except M5, M6, M7, M11 and M12 where the ultrasonic velocity seems to diminish a little. The deviation of the measures is lower in 2011 except in case of M1, M6 and M7. The ultrasonic velocities confirm the observations on degradation. They have not decreased significantly. The variations of the measure for most of the monoliths (except M2 and M9) have diminished. This can be linked to the loss of scales, which induced important measurement variability.

The degradation patterns affecting I Stantari's monoliths (cracks, scaling, granular disintegration, and lichens as biological colonisations) are classical on granites (Silva et al., in Sanmartin et al., 2008). The degradation depends on several intrinsic and external factors. The monoliths have been sculpted in weathered granites as the comparison of the ultrasonic velocities and the observation of degradation patterns on both the near possible extraction site and megaliths shows.

Alvarez and al. (2008) indicate that granular disintegration depends on the rainwater spray and capillarity. Rising damps effects and splashing can also explain the location of the decays, which mainly affect the bottom part of the monoliths. The climate induces differential erosion of the monoliths: the West side suffered from prevailing wind, sunshine and drastic dry- humidity cycles. Degradation leads then to a coarse roughness of the granite surface. Another cause would be the partially buried position of the monoliths (M2, M3 to 5, M7, M8 to M12) before their erection in 1968 (Grosjean's excavations). But none available comparative element could confirm this. More, the presence of saline spray combined to dry periods leads also to the preferential granular and mineral decohesion as the observation and characterisation of powder collected on experimental samples showed. Phyllosilicates, clay minerals and quartz are mainly detached from the surface of the stone in presence of salts, which are supposed to come from marine sprays. Collective (2008) and Sebastian et al. (2008) note that the presence of both soluble salts and swelling clay minerals leads to the scaling.

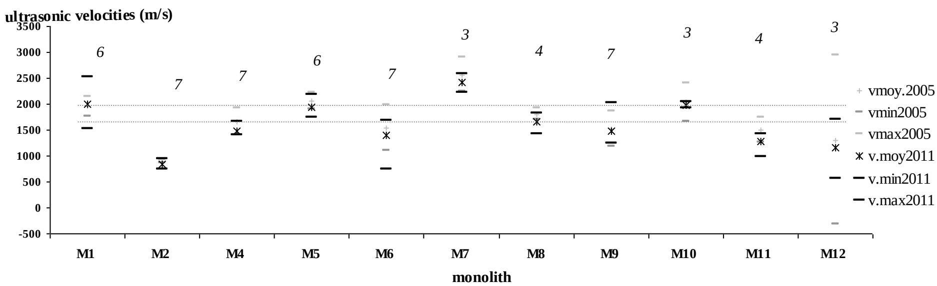

Figure 4. Variation of medium ultrasonic velocities for each monolith from 2005 to 2011. (The grey straight lines correspond to the measurements on the nearest granitic outcrops in 2005. x is the number of measurements that we did along each monolith).

Because the degradation is still active, and because of the lichens development since 1993, one can consider that Casta's consolidation using the Acryl product is no more efficient. More, the diagnosis (Casta, 1988) appeared to be incomplete because the causes of the degradation were not taken into account before this treatment. In another hand, no explanation about the choice of the product was given. Last, the kind and the period of treatment were inappropriate because a rainfall was supposed to wash partially the product (Casta and Poli, undated) and the soil around megaliths is strengthened.

4. Laboratory testing of new consolidants

4.1 Presentation of the studied granite

Because of the difficulties to obtain some Cauria granitic samples coming from the nearby outcrops, experimental samples were cut in damaged area of the Sidobre's granite (near Castres, France). The mineralogy of the Sidobre's monzogranite is closed to the megalithic one. It is constituted in quartz, potassic and calco- sodic feldspars and biotite, which are respectively 36%, 33%, 34% and 6% in volumetric modal composition; from Didier, 1991). Some potassic feldspars are few centimeters in size. The measured ultrasonic velocities are quite equivalent to those coming from I Stantari's monoliths; they vary from 1000 to 3000 m/sec.

50 samples were cut as cubes from 10 cm in size in the peripheral area of the exploited blocks in order to have damaged part of the granite. These areas are weakly

yellowish because of the presence of iron oxides coming from the partial biotite transformation. The granite shows thin fissures.

4.2 Experimental treatments and accelerated ageing protocols

Four consolidant products have been applied on 11 samples each in a classical way of 3 applications until saturation point is reached and using a paintbrush (Begonha and Fojo 2008, Sanmartin and al., 2008):

- The acrylic resin ACRYL™ AC261K as the reference;
- The acrylic resin Paraloïd™ B-72 (Röhm and Haas);
- The ethyl silicate SILRES® BS OH 100 (Wacker) which is largely used in France and seems to give quite good results (eg Alvarez et al., 2008; Beghona and Fojo, 2008; Costa and Rodrigues, 2008; Rojo et al., 2008);
- The same ethyl silicate associated with the water repellent SILRES® BS290 (Wacker) which is a copolymer silane/ polysiloxane without solvent because the use of silanes (Begonha and Fojo, 2008; Wheeler, 2008) and this kind of association seem to give interesting results (Kim et al., 2008).

Both climatic and salt spray accelerated ageing chambers were performed on the samples. 5 samples for each treatment and for one ageing chamber have been studied and 1 other has been kept as the reference. The climatic ageing was performed using a VC4034 chamber from Vötsch. The samples suffered 282 cycles (Table 1). The first 114 cycles reproduced climatic variations closed to South Corse climate ones then temperature constrains were increased during the 168 last cycles.

Cycle 1			**Cycle 2**	
T (°C)	RH (%)	D (h.)	T (°C)	RH (%)
25	65	2	25	65
45	20	2	85	17
10	90	2	10	90
-10	0	2	-10	0

Table1. Details of the climatic accelerated ageing cycle. (T, temperature; RH, relative humidity; D, duration in hours).

Because of the close distance to the sea, soluble salts effects of on strengthened granite had to be investigated. Soluble salt have not been clearly detected except on the surface. Then, non salty samples have been aged using a saline spray chamber (SC450 chamber from Weiss Technik). The samples suffered 210 cycles in presence of halite (NaCl 10% in mass). The protocol of the first 56 cycles was: 2 hours at 25 °C, 5 minutes to increase the temperature until 40°C then the decrease of the temperature during 55 minutes until 25°C. But because of an insufficient drying, the samples were then dried every 6 cycles at 18<T<25°C and 40<RH<65%.

4.3 Laboratory characterisations

The variations of ultrasonic velocities (AU 2000 device) were measured on the three directions perpendicular to the faces of the samples in order to characterize the effects of strengthening and of ageing as well. In order to compare the ageing results, a balancing coefficient has been applied in order to take into account the differences of velocity between the untreated stones.

Weight measurements ($\Delta m = \pm 0.02$ g) were done on samples in order to determine the consumption of the applied consolidant and the loss of material.

The collected powder on the samples was first cleared out of halite using distilled water then crushed. The 0-4 µm granulometric fraction has been sampled. Minerals were identified using a X- Ray Diffractometer D8 Advance from Bruker (Cobalt tube, θ-2θ configuration, 40 KV- 35mA) by way of the powders method and applying the Bragg's law. The nature of clay minerals was also determined by way of oriented deposits, then ethylene glycol and heat (500°C during 2 hours) treatments of the deposits.

4.4 Results

The consumption of the different products is low (Table 2). The products enter weakly in the stones. The variations between the cubes are high in case of acrylic resins.

Consolidant	kg/m²	variations
Acryl	0,027	28,9%
Paraloid	0,010	44,8%
Ethyl silicate	0,145	14,1%
Ethyl silicate+water repellent	0,074	18,2%

Table 2. Average consumption of the consolidants applied on 11 samples.

If we compare the ultrasonic velocity before and after consolidation, it increases for all the treatments. Nevertheless, the strengthened treatment induces variations in terms of ultrasonic velocity (Figure 5):

- the velocity increases more in presence of consolidant containing ethyl silicate than in presence of acrylic resins;
- the treatment led to differences depending on the direction of measurement (parallel or perpendicular to the foliation);
- Acryl treatment shows the lowest increase of velocity.

After the climatic ageing, all the ultrasonic velocities are reduced and are quite equivalent, excepted Paraloïd. The strengthened treatments seem to have a little residual effect because the difference is a little bit lower than in case of untreated stones.

In the case of saline spray ageing, all the treated stones show a positive difference of the velocities between before and after while this difference is negative in case of untreated ones. The best results concern the ethyl silicate- base ones.

Powder appeared on cubes after ageing. Very few grains were collected using a paintbrush on the samples which suffered the climatic ageing. The powder was collected in the case of saline spray ageing. It was weighted after dissolution of halite in order to determine the contribution of the stones. Table 3 recapitulates the results. Untreated cubes loss more material than the treated ones. The ageing of the treated cubes with ethyl silicate+ water repellent treatment led to a quite important loss of material compared to the other treatments. Paraloïd B72 and Ethyl silicate are the less affected.

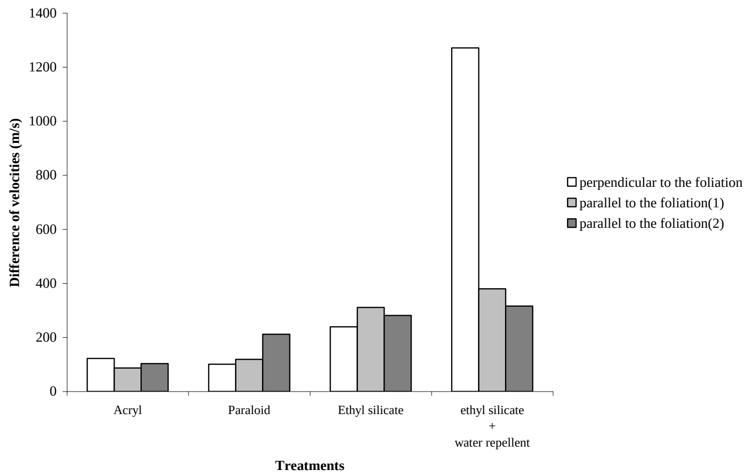

Figure 5. Evolution of the ultrasonic velocity depending on the nature of the applied treatment

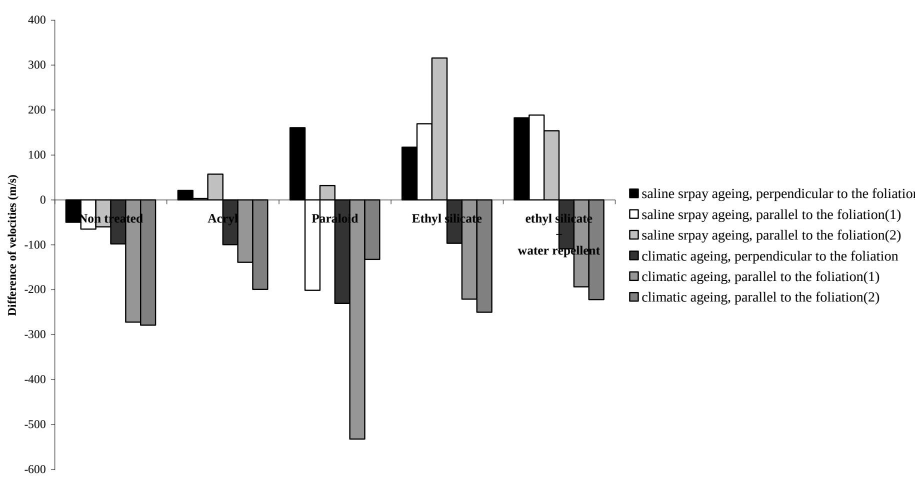

Figure 6. Comparison of the ultrasonic velocity variations vs the applied experimental ageing

The collected powder is mainly composed of biotite, chlorite, quartz and feldspar. Interstratified mineral containing a swelling clay mineral has been detected in very few quantities. This is an illite- smectite mixed layer if we consider the XRD positions of dome- shaped peak depending on the preparation (1.28 nm deposit, 1.33 nm ethylene glycol treated, 1.23 nm (Ir weak) heated). The content of smectitic layer is low.

The accelerated climatic ageing affects all the strengthened cubes. The differences between the products are not significant. Ethyl silicate seems to give more resistance to the stones than acrylic ones face to a saline spray accelerated ageing. But none treatment gives satisfaction as the equivalent ultrasonic velocities measured on treated and untreated samples suggested.

consolidant	climatic	Saline spray (g)
Untreated	few grains	0,57 ± 0,04
Acryl	*Id.*	0,05 ± 0,04
Paraloid	*Id.*	0,018 ± 0,04
Ethyl silicate	*Id.*	0,016 ± 0,04
ethyl silicate+water repellent	*Id.*	0,21 ± 0,04

Table 3. Collected mass (g) of powder on aged cubes depending on the nature of the treatment and the kind of experimental ageing they suffered.

The collected powder on samples after ageing under salt spray contains more phyllosilicates and quartz in the Paraloïd sample than in the Ethyl silicate sample. This shows a higher alteration of the Paraloïd strengthened samples.

The measured consumptions of all the experimental samples are low. In another hand, the increase of the ultrasonic velocity after treatment was weak and not so different from the velocities measured on untreated weathered granite. Therefore the penetration depth is probably low because of the porosity (Sanmartin et al., 2008). A better evaluation of the durability of these kind of treatments applied on a granite needs then a higher penetration depth. This last would also favour a better resistance to hygroscopic salt degradation (Sanmartin et al., 2008). Despite the employed application technique of the products is the usual one (eg Begonha and Fojo, 2008) and the most adapted to the isolated sites, the application method must be modified. Different techniques can be applied: immersion gives good results in laboratory (Sanmartin et al., 2008) but it is totally not adapted to such big and heavy artworks the monoliths are; the gravity impregnation did not lead to good results on I Stantari's megaliths because the acrylic resin did not penetrate enough in the porosity. Last, vacuum impregnation as the Ibach method could be a good way for both in field and in laboratory treatments.

The two families of resins are different in terms of their properties. Acrylic resins have quite elastic properties compared to ethyl silicate ones but they are film- forming. They do not give good results in terms of durability. The first results concerning ethyl silicate and the combination of ethyl silicate with silane are not also convincing. Silicate- based resins are not elastic and can induce crack networks in the resin and the treated stone as well. The presence of clay minerals increases the issue (Félix, 1995) but the addition of micro or nano- particles of silica seem to be a good way of investigation according to Escalante et al. (2000) and Kim et al. (2008).

5. Conclusions

The strengthening treatment applied in 1993 did not give any satisfaction if we consider the evaluations done in 2006 and 2011. In order to find a durable and efficiency consolidant, treatments tests have been performed taking into account the environment (climate and marine environment), the nature of the cultural heritage objects (which do not allow every kind of treatment application) and the degradation state of the granite. They are also unsatisfactory because they do not increase the resistance of the stone in front of accelerated ageing. The reason seems to be inherent to the employed technique to apply the products. The consumption was too low and the products should penetrate deeply to ensure a better efficiency. A quite low porosity and supposed weak capillary properties lead to the difficulty to treat such kind of materials.

New experimental tests are therefore needed to know if it is possible to treat on site I Stantari's monoliths using products which would be efficient through time. Vacuum impregnation could be a good way to test them even this treatment is difficult to perform in such archaeological sites. In front of their apparent inadequacy over time, some other strengthened products such as nano- consolidant products must be then studied.

Complementary tests and analyses such as experiment on samples containing soluble salts, pores distribution and capillarity study, thermal conductivity and spectrocolorimetry will be also perform before and after climatic and saline spray experimental ageing. All the results will be able to precise what the best conservation surveys are for each monolith including a device against rising damp.

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