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Critical Degree of Saturation as a Threshold Moisture Level in Frost Weathering of Limestones

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ABSTRACT

The moisture content has a deciding impact on the behaviour of a rock undergoing frost action. A particular critical degree of saturation S_{cr} characterizes each material; only when the moisture content exceeds S_{cr} will the material be damaged by frost. This parameter was defined for ten French limestones by measuring their dynamic Young's modulus. S_{cr} values depend on porosimetric characteristics of the rocks, especially their trapped porosity. The critical degree of saturation accounts for the various rock dilatometrical behaviours during a freeze–thaw cycle. © 1997 by John Wiley & Sons, Ltd.

RÉSUMÉ

Le degré d'humidité d'une roche a une importance déterminante sur son comportement lorsqu'elle est soumise au gel. On peut définir pour chaque matériau un degré de saturation critique S_{cr} au-dessus duquel le gel causera des dommages. Ce paramètre S_{cr} a été défini pour dix calcaires français en effectuant des mesures du module dynamique de Young de ces roches. Les valeurs des paramètres S_{cr} obtenus dépendent des caractéristiques des roches et notamment de leur porosité piégée. Le degré de saturation critique, allié aux caractéristiques porosimétriques, permet d'expliquer les divers comportements dilatométriques de ces roches au cours d'un cycle de gel–dégel. © 1997 by John Wiley & Sons, Ltd.

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KEY WORDS: frost weathering; critical saturation; dynamic Young's modulus; limestones

INTRODUCTION

Rock frost sensitivity depends on lithological and environmental parameters. The main lithological parameters seem to be porosity (McGreevy, 1982; Lautridou and Ozouf, 1982), porosimetry (Bousquie, 1979; McGreevy, 1982; Letavernier, 1984; Remy, 1993), specific surface area (Hudec, 1979; Remy, 1993) and permeability (Bousquie, 1979; Lautridou and Ozouf, 1982; Letavernier, 1984). The environmental parameters commonly taken into account in the freeze–thaw experiments carried out in the laboratory are: methods for saturating samples; degree of saturation; cooling rate; temperature and duration of plateaux, especially for the plateau at minimal temperature (McGreevy, 1982; Letavernier, 1984; Stark, 1989; Weiss, 1992; Remy, 1993).

Among these parameters, the degree of saturation is certainly the most important. "A unanimous

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conclusion is that for the operation of frost shattering, an adequate moisture supply is indispensable" (McGreevy, 1982). Rock water contents in cold environments are usually neither close to saturation (water supply is insufficient for this: White, 1976; McGreevy, 1982; Hall, 1986; 1991; 1995) nor close to a dry state (drying in natural conditions does not cause all the adsorbed water to evaporate: Prick, 1996). The degree of saturation depends not only on the way the sample is hydrated (sudden or gradual immersion; at normal atmospheric pressure, under vacuum or under pressure; etc.) and on the duration of this immersion, but also on the porosimetry of the rock. The highest saturation that can be reached by immersion at normal atmospheric pressure depends on distinct porosimetric characteristics, including the capillary rise coefficients, the pore interconnection degree, the occurrence of trapped macropores, and so on (Bousquie, 1979; Remy, 1993).

In connection to this relation holding between a sample degree of saturation and its lithological characteristics, it is possible to define for each type of rock a degree of critical saturation S_{cr}: with degrees of saturation greater than or equal to S_{cr}, a rock will be damaged by frost, according to former work (Fagerlund, 1971). S_{cr} values are specific for each material. The results discussion will show that there are some limitations in the applications of the S_{cr} parameter results.

METHOD

The degree of critical saturation is determined by measurements at various degrees of saturation of a particular parameter, like the material dilation behaviour (Mamillan, 1984), its modulus of elasticity (Fagerlund, 1971), which can be determined by measuring ultrasonic wave propagation (Remy, 1993) or with the fundamental vibration frequency (Weiss, 1992). In our experimental work, we determined the fundamental vibration frequency by the Grindosonic apparatus.

The modulus of elasticity (or Young's modulus) expresses the rock stiffness, as it measures the stress required on an elastic material to produce a specific deformation. These modulus values vary strongly with the rock grain sizes, their interconnection degree and the porosity: the higher the modulus value, the smaller the material deformation when it undergoes a strain (Allison, 1987; Weiss, 1992; Remy 1993). As the modulus of elasticity reflects this porosimetric parameter, and thus the microcracks formation, it is considered as a tool for the weathering evaluation (Weiss, 1992).

The Grindosonic apparatus uses the principle that elasticity theory can be applied to materials by directly measuring the fundamental vibration frequency of a rock sample of prescribed dimensions following shock excitation. Samples are struck to set up a mechanical vibration pattern, rather than being subject to continuous flexure as in the most widespread mechanical strength tests. The vibration pattern is converted into an electronic signal via a piezo-electric detector held in contact with the test pieces (Figure 1). The signal is amplified by the apparatus before being fed to the instrument input. After attenuation of initial spurious wave patterns which have complex harmonics, the time for eight waves to pass is measured. The oscillation period is then automatically established. Young's modulus is calculated with quite good accuracy by the Grindosonic on the basis of a material fundamental vibration by inputting some data like specimen shape, weight and density. The Grindosonic apparatus also makes it possible to measure torsion, which can lead to the determination of shear modulus G: that modulus reflects the resistance of a specimen to deformation when subjected to shear stress.

Test pieces to be used can be cut to a variety of shapes including bars, cylinders and disks. Specimens must have a thickness ratio of more than two.

The Grindosonic provides a non-destructive determination of rock strength, which is an advantage in our experiments: individual samples can be submitted to repeated measurements during a series of freeze–thaw cycles in order to identify consequent changes in material characteristics. The result's accuracy is entirely satisfactory in weathering simulation experiments: measurements are made quickly and easily, and are accurately reproducible as long as specimens are well shaped and exposed before measurements to stable atmospheric conditions (temperature and water content) (Weiss, 1992). This apparatus has been used in several geomorphological works: frost and salt weathering experiments (Weiss, 1992), weathering by fire (Goudie *et al.*, 1992), slope stability and strength evaluation in the field (Allison, 1988; 1990). According to Allison (1990), the Grindosonic apparatus gives better results than other more widespread techniques, like the Schmidt hammer or the triaxial Hoek cell (standard laboratory

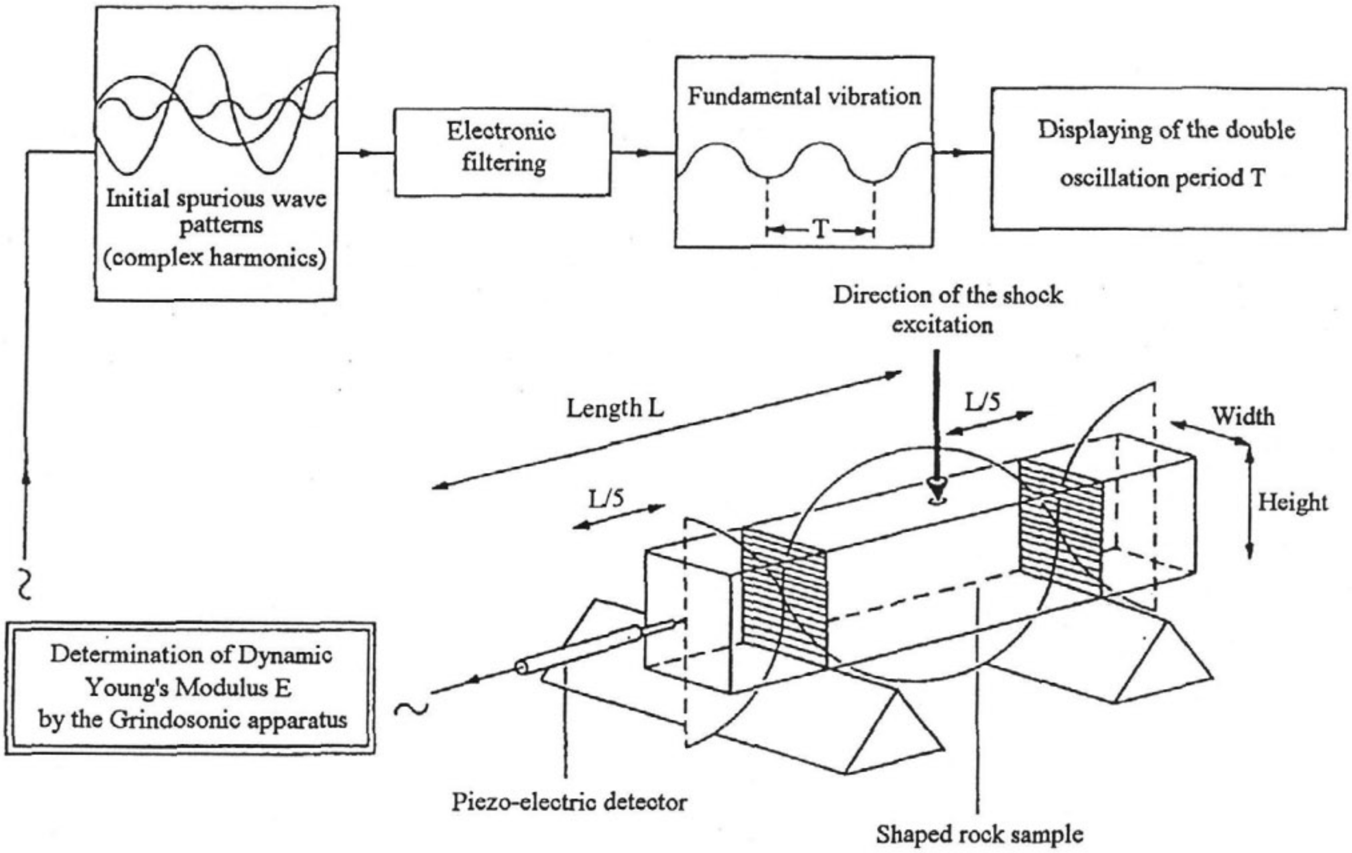

Figure 1 Fundamental flexural vibration of a square shape bar analysed by the Grindosonic apparatus (after Weiss, 1992).

procedure testing material failure over a range of confining pressures).

According to Allison (1988), elastic properties are the most appropriate ones for evaluating rock strength for a geomorphological understanding of rock behaviour. Cooks (1983) notes that elasticity may be the most important factor in determining the behaviour of rocks being weathered. The decrease in Young's modulus values has been successfully considered as a reflection of weathering action in former geomorphological work (Allison, 1988; 1990; Goudie *et al.*, 1992; Weiss, 1992). Nevertheless, other researchers consider that the most useful index of rock strength is the tensile strength of the rock (Fukuda, 1983) or the Quality Factor (based on damping measurements of the fundamental transverse frequency; Janssen and Snyder, 1994), or even that the elasticity modulus is not a reliable parameter (Hallet, personal communication).

It is clear that, although such mechanical properties measurements have rarely been used by geomorphologists, they are very widely used by engineers in studies of frost damage of concrete (Neville, 1981).

SAMPLING AND LABORATORY TESTING

Results are presented here for tests conducted on ten Jurassic or Cretaceous French limestones. These rocks are used in France as building stones. They were chosen because of the variety of their physical characteristics, especially their porosimetry (Table 1) and the availability of other petrographic or weathering studies on these rocks (Bousquie, 1979; Letavernier, 1984). The limestone porosimetry analysis provides a classification based on a dispersion coefficient C_d (that reflects the porosimetric spectrum spreading) and the pore classes number (strictly unimodal, spread unimodal, bimodal, multimodal) (Remy, 1993).

Cylindrical specimens (11 cm long, 4 cm in diameter) were cut perpendicular to bedding. Once prepared, the samples were washed, dried at 50 °C to constant weight and left to cool in the open air before measurements with the Grindosonic. The critical degree of saturation can be determined by freezing porous material specimens with various degrees of saturation and by comparing the Young's modulus values after freezing (E) and in a fresh state (E_0). An example of a determination

Table 1 The physical characterization and degree of critical saturation of ten French limestones.

Rock	Porosity	C_d	N_W	MPD	N_p	H	E	S_{cr}
Comblanchien	Strictly unimodal	0.89	1.30	0.015	0	97.53	76	100
Charentenay	Spread unimodal	1.07	29.80	0.875	11.66	87.86	17	88
Larrys		1.15	20.92	0.579	4.61	93.45	26	93
Tervoux		1.51	25.56	0.702	8.98	90.77	15	90
Vilhonneur		1.56	9.15	0.183	8.64	89.87	45	83
Tercé		1.70	21.31	0.805	7.82	91.52	19	90
Lavoux		2.22	25.13	0.736	16.61	84.33	21	90
Caen	Multimodal	4.08	31.42	1.180	19.66	84.23	12	60
Sireuil	Bimodal	23.89	31.49	17.64	28.56	71.55	8	58
Brézé (tuffeau)		29.60	47.23	2.58	17.02	84.34	3	92

C_d is dispersion coefficient. Reflects the porosimetric spectrum spreading: $C_d = (P_{80} - P_{20})/P_{50}$, where P_{80}, P_{50} and P_{20} are the mercury injection pressures corresponding to an invasion of 80, 50 and 20% of the porous media. The C_d values allow the characterization of rock porosity as unimodal (with only one pore class, narrow or large), multimodal (with several pore classes) and bimodal (with two pore classes) (as defined in Remy, 1993).

N_W is total porosity: porosity measured perpendicularly to bedding, by immersion under vacuum (%).

MPD is median pore diameter, measured by Hg porosimetry on the basis of pore volumes·(μm).

N_p is trapped porosity as a percentage of total porosity N_W. The trapped porosity is defined as the discrepancy between N_W and H

H is Hirschwald coefficient or saturation coefficient (part of the porosity N_W that is accessible to water in 48 hours) (%).

E is mean dynamic Young's modulus of the fresh rock (GPa).

S_{cr} is value of the critical degree of saturation (%).

of S_{cr} for the Tercé is shown in Figure 2. Each dot corresponds to one specimen wetted by progressive immersion under atmospheric pressure and afterwards dried to a specific moisture content. The sample is then frozen rapidly and thawed for eight cycles following experimental conditions adopted by Weiss (1992), i.e. 6 hours cycles between +20 °C and −30 °C, with a maximal rate of cooling of 0.8 °C/min between several temperature plateaux of 30 minutes in duration (at +20, +10, −1, −10, −20, −30, −10, +20 °C). The degree of saturation remains constant during the experiment: all the rock cylinders are sealed in plastic foil after wetting by simple immersion at atmospheric pressure. A nick-point in the diagram of dynamic Young's modulus versus saturation can be seen easily. This nick-point defines the values of S_{cr}: with higher saturation, weathering takes place and can be observed as a

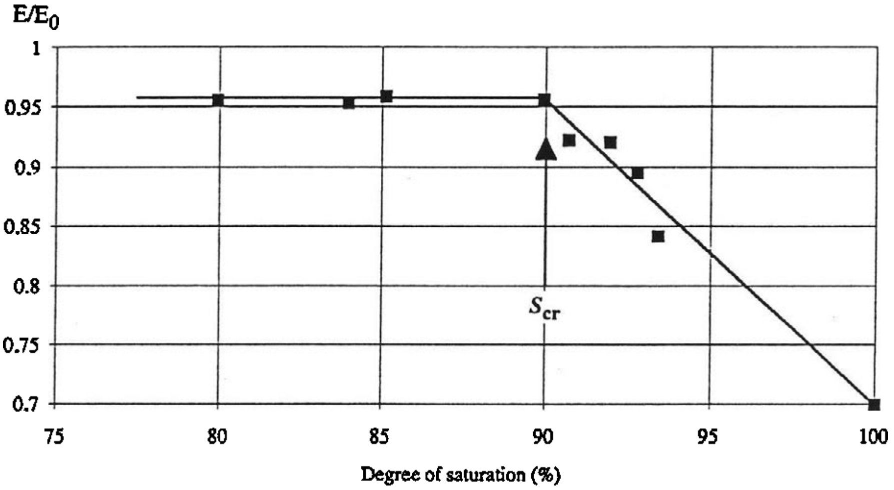

Figure 2 Determination of the critical degree of saturation S_{cr} of the Tercé limestone. The nick-point on this diagram defines the value of S_{cr}: by freezing with higher degree of saturation, Young's Modulus value E decreases significantly.

Young's modulus decrease. For saturations greater than S_{cr}, a linear relation exists between the weathering rate and the saturation degree.

This experimental approach has been used in other works (Fagerlund, 1979; Houiou, 1979; Mamillan, 1979; Weiss, 1992), mainly by engineers working on concrete. According to Fagerlund (1979), the number of freezing and thawing cycles seems to have no or very little effect on S_{cr} values. Weiss (1992) does not share this opinion. He observes a decrease if the S_{cr} value when the cycle number increases (from 7 up to 50): rock is weathered by frost with lower moisture content after undergoing former freezing cycles, even if these cycles taken separately were poorly aggressive.

Rock frost behaviour was observed using two complementary techniques: accurate dilation measurements during one freeze–thaw cycle (Prick *et al.*, 1993; Pissart *et al.*, 1993; Prick, 1995) and Grindosonic testing (fundamental vibration frequencies) of rock samples after four successive frost sequences. Results of these two experimental procedures were compared, as explained below. It was impossible to carry out both measurements on the same samples, because dilation experiments require boring holes and fixing plastic bases that prevent the calculation of Young's modulus values. Nevertheless, both measurements were carried out on the same ten limestones.

Dilation measurements of rock subjected to freezing and thawing conditions have rarely been carried out (Matsuoka, 1988; Weiss, 1992). They are used in geomorphology to examine the frost behaviour of porous rocks, including unfrozen water migration (Prick *et al.*, 1993; Pissart *et al.*, 1993; Prick, 1995). This migration at negative temperature is responsible for anisotropic strains of the samples and for important changes in the water distribution inside the rock; the main dilatometric results obtained in former work will be summarized below. Two variations in length and one variation in diameter of rock cylinders are measured using electronic displacement transducers, with an accuracy of ± 5 μm. These measurements and rock temperature measurements are automatically carried out during one freeze–thaw cycle performed in a climatic simulator (between +20 °C and −20 °C, with a rate of variation in temperature of 0.5, 2 or 10 °C/h, and a 24 hour plateau at minimal temperature).

Young's modulus testing was performed on four samples of each rock type, submitted to the same cooling conditions as the samples submitted to dilation experiments (see Table 4).

RESULTS

S_{cr} Evaluation

The S_{cr} values obtained range from 58 to 100% (Table 1). Among mechanical and physical parameters, the trapped porosity N_P has the highest correlation with S_{cr} (Table 2). When the best five correlated parameters (C_d, N_W, MPD, N_P, E) are involved in a multiple correlation, 89.5% of the S_{cr} values can be accounted for.

A rock S_{cr} value is thus determined more by porosimetry, and especially trapped porosity, than by total porosity. Trapped porosity results from air bubbles being trapped in the porous medium during wetting at atmospheric pressure. The wetting kinetics is strongly influenced by pore dimensions and distribution. As capillary suction forces are more developed in narrower pores, the pores that get trapped and filled with air are macropores; the hydrodynamic role of these bubbles is important (Bousquie, 1979). Rocks characterized by a large porosimetric spectrum (bimodal or multimodal rocks) are more susceptible to the air trapping process. Hence, their trapped porosity (N_P) is higher and their saturation coefficient after 48 hours (H) is lower. On the other hand, unimodal rocks undergo a more homogeneous capillary rise: N_p values are limited and H values are higher.

Frost sensitivity is sensitively dependent on macroporosity. In the case of a saturated porous medium, large pores act as water accumulation centres in which stresses approaching the strength of the material can build up in concentration with the transformation of water into ice (Neville, 1981). But the water content levels that are the most common in nature are far from complete saturation (White, 1976; McGreevy, 1982; Hall, 1986; 1991; 1995); in that case, macroporosity protects rocks from slow freezing by allowing ice extrusion to empty spaces (McGreevy, 1982) or, more likely,

Table 2 Simple correlation coefficients of some mechanical and physical parameters with the critical degree of saturation S_{cr}.

Dispersion coefficient	C_d	0.33
Total porosity	N_W	0.34
Median pore diameter	MPD	0.67
Trapped porosity	N_P	0.82
Saturation coefficient[1]	H	0.79
Dynamic Young's modulus	E	0.45

[1] H is highly correlated with N_P ($r = 0.98$) and was not taken into account in the multiple correlation.

unfrozen water migration towards macropores that are partly filled with water and in which freezing can begin at higher temperatures than in micropores (Prick, 1995).

Moreover, macroporous rocks are more permeable than microporous ones (in which capillary forces are strong enough to prevent water from moving through the porous medium) and can be subjected to unfrozen water migration at the scale of the whole sample, from the central part to the outer colder part of the sample, where ice lenses are formed (Weiss, 1992; Prick, 1995), which is comparable to the cryosuction process in soils (Walder and Hallet, 1986).

This accounts for the good correlation between trapped porosity and the critical degree of saturation. Nevertheless, mechanical strength is also important and may explain the low S_{cr} values of Caen and Sireuil limestones.

Comblanchien limestone has a very low porosity (made up of micropores) and a high mechanical strength; this rock is not weathered by frost when wetted by immersion, but becomes frost sensitive only after saturation under vacuum. This confirms Lautridou and Ozouf's (1982) observations: rocks with a total porosity lower than 6% are not frost sensitive.

It is important to underline that the S_{cr} values obtained for the tested rocks cannot be used without some precautions in field weathering studies. As the behaviour of rock samples subjected to freezing is highly dependent on thermic conditions (rate of cooling, minimal temperature, number of cycles, etc.) it is clear that diverse freezing conditions influence the relative importance of water migrations and volumic dilation, the effectiveness of the weathering, and hence the value of S_{cr}. The influence of the cycle number has already been emphasized (Weiss, 1992). Nevertheless, the S_{cr} values obtained during our work were very useful and allowed a correlation with other experimental results (see below), even if S_{cr} evaluation and dilation measurements were not carried out in the same thermic conditions. But it is necessary to draw the readers' attention to possible oversimplified views of the critical degree of saturation: this is not an absolute value directly applicable to studies of frost weathering in nature.

Connection with Dilation and Mechanical Behaviour

The critical degree of saturation was really useful for understanding the dilation and mechanical behaviour of limestones undergoing various experimental conditions. It has been possible to classify the observed behaviours by combining porosimetric characteristics (multimodal porosity, unimodal porosity, poorly porous rocks) and degree of saturation (higher or lower than S_{cr}). Table 3 presents the final classification results, which are based on frost weathering scales developed by other authors (Mamillan, 1979; Weiss, 1992; Remy, 1993).

The main results from former dilatometric work on frost action (Prick *et al.*, 1993; Pissart *et al.*, 1993, Prick, 1995) show that it is only when rock samples are close to saturation and submitted to a relatively rapid cooling that a large increase in volume is observed, in connection with the 9% volume expansion effect induced by the transformation of water into ice (e.g. a 10 cm long Brézé cylinder with a saturation coefficient of 98% and submitted to a 2 °C/h cooling shows a maximal length expansion of 70 μm). For samples relatively far from a saturated state, volume can diminish during freezing, most often in an anisotropic way. This contraction is very commonly shown by rocks with a multimodal or bimodal porosity (Tables 1 and 3); it is due to unfrozen water migration at negative temperatures, according to the well known process of progressive ice accretion and ice lenses formation (Walder and Hallet, 1986; Hallet *et al.*, 1991; Weiss, 1992). This process has also been proven by the changes in the water distribution inside the rock as observed after thawing (Prick, 1995). For example, a Brézé cylinder with a saturation coefficient of 72% shows in the dilation curves a maximal length contraction of 50 μm when submitted to a 2 °C/h cooling.

The unfrozen water migrations and the cryosuction process mentioned above are due to two processes, identified in the hydraulic pressure theory (hydraulic and osmotic pressures generated by ice formation induce water migration ahead of the ice front: Powers, 1945; Fagerlund, 1979) and in the capillary pressure theory (water migrations are induced between pores of various radii, in which water freezes at different temperatures: Everett, 1961; Remy, 1993). Moreover, thermic contraction also occurs during the experiments, but is usually masked in the dilation curves by the volume expansion.

A sample with unimodal porosity also swells when highly wetted because of the transformation of water into ice. But with degrees of saturation lower than S_{cr}, no contraction is observed: unfrozen water does not migrate here as well as in a

Table 3 Frost behaviour of the limestone samples, according to dilation behaviour and dynamic Young's modulus variations.

Rocks	Mechanical strength evolution (dynamic Young's modulus: see Table 4)	Dilatometrical behaviour when degree of saturation is:	
		higher than S_{cr}	lower than S_{cr}
Rocks with a multimodal or bimodal porosity			
Brézé	Sensitive to all freezing conditions	Strong dilation	Contraction
Sireuil	Sensitive to all freezing conditions except the 0.5 °C/h frost		
Caen	Not sensitive to any tested freezing conditions.		
Rocks with a unimodal porosity			
Lavoux Charentenay Tercé Tervoux	Sensitive to freezing in saturated conditions (at atmospheric pressure)	Dilation	Complex and Anisotropic Behaviour
Larrys Vilhonneur		Dilation	Stability
Poorly porous rocks			
Comblanchien	Not sensitive to any tested freezing conditions	Insignificant dilatometrical response, even after saturation under vacuum	

multimodal porous medium. As water can only migrate between micropores and adjacent macropores, little or no water redistribution occurs at the larger sample scale. Dilatometrical variations are nil in rocks with a low porosity (Larrys, Vilhonneur) and they are anisotropic and complex in other rocks (Lavoux, Charentenay, Tercé, Tervoux).

Comblanchien limestone is not frost sensitive: it absorbs so little water that its dilation behaviour is insignificant, even if it is saturated under vacuum.

These observations clearly show that rock frost behaviour depends on lithological parameters and on environmental parameters (among which the degree of saturation is the most important, much more important than the cooling rate, for example). Nevertheless, it is impossible to associate each type of dilation response to a mechanical weathering evaluation. The decrease of Young's modulus values after one freeze–thaw cycle (Tables 3 and 4) clearly indicates that there is no direct relation between, for example, a contraction behaviour and a lack of shattering effect (Brézé limestone weathers under all four experimental conditions; Caen limestone weathers under none of the experimental frost). The exact role of mechanical properties from a general point of view can hardly be measured, but this certainly explains discrepancies between these limestones, frost sensitivity and Remy's (1993) weathering scale. According to that scale, multimodal rocks are less frost sensitive than unimodal ones, which represents results opposite to ours. On the other hand, our results confirm Letavernier's (1984) frost sensitivity scale, which is based only on the porous media characterization and not on mechanical parameters.

CONCLUSIONS

The critical degree of saturation is a key factor in understanding frost weathering. When particular thermic conditions are considered, this parameter reflects the influence of lithological characteristics on the frost sensitivity and defines the part of the porous medium that has to be free of water in order not to build up a breaking strain. So, it acts as a threshold between two main types of frost: frost occurring with a high saturation level which leads to a sample dilation response and to significant irreversible damage by bursting (Ozouf, 1983; Letavernier and Ozouf, 1987), and frost occurring with moisture content lower than S_{cr} which leads to anisotropic contraction or dilatometrical stability and to a scaling process due to the formation of segregation ice (Ozouf, 1983; Walder and Hallet, 1986; Hallet *et al.*, 1991).

Table 4 Mean decrease (%) of the dynamic Young's modulus *E* for each type of limestone undergoing one freeze–thaw cycle under four different experimental conditions.

Moisture content:	saturation	75% of full saturation		
Cooling rate:	2 °C/h	10 °C/h	2 °C/h	0.5 °C/h
Brézé	12.87	11.19	5.72	7.6
Sireuil	6.26	5.37	2.71	0
Caen	0.19	0	0	0
Lavoux	2.66	1.34	0.27	0.94
Charentenay	2.79	1.04	0.55	1.23
Tercé	11.19	0.71	0.03	1.31
Tervoux	9.25	1.13	0	1.95
Larrys	9.65	0.64	0.64	0.48
Vilhonneur	2.86	0.53	0.19	1.1
Comblanchien	1.14	0.6	0.7	0.71

The results provide the basis for a frost sensitivity scale that concurs with dilation results obtained previously (Prick *et al.*, 1993; Pissart *et al.*, 1993; Prick, 1995). Nevertheless, this does not allow a direct and full understanding of limestones' frost sensitivity, except if the rock mechanical strength is taken into account. Frost sensitivity results obtained in this work are confirmed by weathering studies carried out with other experimental methods (Letavernier, 1984).

In conclusion, the critical degree of saturation is a rock parameter that can be evaluated easily with the Grindosonic apparatus. It can provide invaluable information in frost weathering studies and it will most probably be more widely used in the future, not only by engineers but also by geomorphologists.

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