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Investigation of the Freezing—Thawing Effect on the Slip Resistance of Natural Stones †

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Abstract: The effect of freeze–thaw cycling on the slip resistance of dimension stones was investigated. Slip and frost resistance of limestones, granites and marbles were determined via pendulum tester in dry and wet conditions and controlled freeze–thaw cycles, respectively. Unpolished surfaces under dry conditions (mainly granites and marbles) were positively affected by freezing-thawing. In wet surfaces no significant change was observed. Polished surfaces were not affected even after 100 freeze–thaw cycles. Electron microscopy showed increased wear, hence roughness, of unpolished surfaces after freezing–thawing; homogeneity of polished surfaces prevented slip resistance from being significantly affected.

Keywords: slip resistance; frost resistance; dimension stones; unpolished surfaces; polished surfaces

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1. Introduction

Slip resistance is the property of a floor surface to maintain the adhesion of pedestrian footwear and it has become a considerable issue in the design of dimension stones used as floor coverings. The assessment of anti-slip characteristics of the flooring materials is of major importance in order to prevent slipping accidents and ensure safe movement. The characteristics of both the footwear and the walking surface, the temperature and other environmental parameters, such as water, moisture, ice, dust, etc., are important factors that affect slip resistance. Slip resistance measured for polished materials is obviously lower than that obtained from other surfaces, especially in wet environments.

Wetting and drying and freezing and thawing have proven to be important physical processes affecting physical mechanical properties of natural stones, which have been investigated by several researchers, e.g., [1–3]. However, studies concerning the influence of these factors on the slip resistance of natural stones are very few focusing mainly on the investigation of other affecting parameters, such as floor roughness, liquid viscosity and shoe materials [4], or floor surface finishes [5].The effect of mineralogical and petrographic characteristics, chemical properties, physical and mechanical properties, the surface finish, and its dryness or wetness on the slip resistance were identified by Çoşkun et al. [6]. Therefore, this study investigates any potential effect of freezing–thawing on the slip resistance of dimension stones for flooring considering their surface type as well as the impact of wet or dry environment. This effect could be expressed by the ratio of free surface area (FSA) versus nominal surface area (NSA), which was defined as the specific surface area (SSA) of each stone specimen examined, given in Equation (1):

$$SSA = \frac{FSA}{NSA} \geq 1, \quad (1)$$

FSA is the real surface area of the specimen, which is susceptible to wear by freeze–thaw cycles. NSA is the area of the rectangle, which is equal to length times width. The magnitude of SSA is ideally equal to 1, when FSA of the specimen has no porosity, no cavities, no veins, or anything else that results in a rough surface. It is obvious that SSA can be increased due to freeze–thaw action resulting in less slippery surface.

2. Materials and Methods

A total of 2 limestones (L1, L2), 2 granites (G1, G2), 2 calcitic (C.M.1, C.M.2), and 2 dolomitic marbles (D.M.1, D.M.2), which are frequently used in various construction projects as flooring materials, were chosen to be laboratory tested in this research. Six specimens from each type of stone were appropriately prepared in order to perform the slip resistance and frost resistance tests at LITHOS, the ornamental stones quality control laboratory of the Hellenic Survey of Geology and Mineral Exploration (HSGME), according to the applicable EN Standards, EN 14231 and EN 12371, respectively. Specimens were prepared in the form of rectangular prisms with dimensions 13.6 cm $\times$ 8.6 cm $\times$ 2.0 cm (length, width, and thickness).

2.1. Slip Resistance Test

Slip resistance testing was performed according to EN 14231:2003 [7], through a pendulum friction tester incorporating a rubber slider. This tester provides the slip resistance value (SRV), that is the friction between the slider and the test surface, and should be measured both in dry (SRV_d) and wet (SRV_w) conditions. For the measurements under wet conditions the specimens were immersed in tap water for at least 2 h prior to testing; before each swing of the pendulum, the test surface and the slider were thoroughly wetted.

2.2. Freeze–Thaw Cycles Testing

For the freeze–thaw cycling tests, all specimens were initially saturated by submerging them in tap water for at least 48 h and then placed in the freeze–thaw chamber, according to EN 12371:2010 [8]. Each cycle consisted of a 6 h freezing period in air from +17 °C to −12 °C, followed by a 6 h thawing period from −12 °C to +17 °C during which the specimens were immersed in water. Specimens were initially subjected to 50 cycles and then optically examined for their integrity. After that, slip resistance was determined according to the procedure described in Section 2.1. Then, specimens were exposed to another 50 cycles of freezing–thawing and their final slip resistance was determined.

3. Results

The lowest slip resistance values (SRV) were measured for the limestone specimens (L1, L2). Test results revealed that SRV_w range between 33 and 40 for unpolished limestone specimens, which are almost ultimate values for flooring application, as proposed in the appropriate specification EN Standards (SRV > 35), and 3 to 4 for the polished ones, as illustrated in Table 1. This indicates that freeze–thaw action has not any significant impact on the roughness of these specimens, although in the case of unpolished specimens a mild trend of decreasing is observed, being attributed to the calcitic content of limestones and water action. Furthermore, SRV_d measured for the unpolished specimens (before and even after 100 cycles) ranges between 63 and 68, indicating that surfaces were not practically affected by freeze–thaw action; this is also clear among SRV_d measured for the polished specimens.

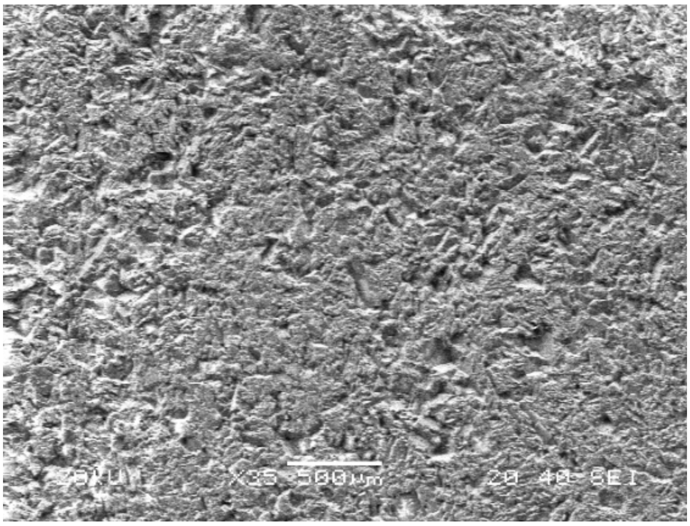
Results concerning unpolished marble and granite specimens demonstrated an increasing trend of SRV_d after their subjection to frost resistance testing, especially after 100 cycles. Scanning Electron Microscopy (SEM) analysis indicated widely distributed surface anomalies on the unpolished specimens making them more susceptible to wear caused by freeze–thaw action, thus increasing their roughness and rending them more slip-resistant. As far as polished surfaces are concerned, Table 1 shows a slight increase of roughness in the case of granite, that may be attributed to its multi-mineral composition, while marbles seem to be practically unaffected by freeze–thaw action.

Table 1. Average SRV before and after freeze–thaw cycling.

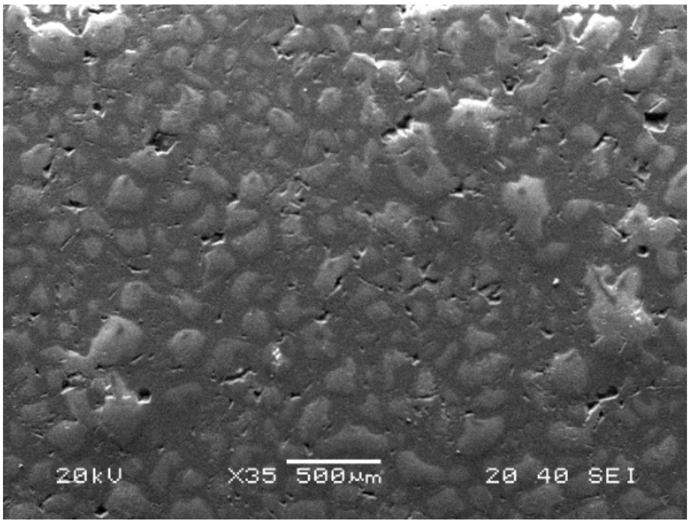
Material	Dry Conditions						Wet Conditions					
	Unpolished			Polished			Unpolished			Polished		
	SRV (before)	SRV (50 cycles)	SRV (100 cycles)	SRV (before)	SRV (50 cycles)	SRV (100 cycles)	SRV (before)	SRV (50 cycles)	SRV (100 cycles)	SRV (before)	SRV (50 cycles)	SRV (100 cycles)
Limestone 1 Limestone 2	63	67	68	37	39	35	40	34	33	3	4	4
Granite 1 Granite 2	71	83	86	46	49	54	52	50	49	4	5	5
Calcitic marble 1 Calcitic marble 2	70	88	94	38	47	39	66	60	61	6	6	5
Dolomitic marble 1 Dolomitic marble 2	74	89	93	49	53	49	64	63	64	9	8	6

Thin sections of the unpolished and polished surfaces of marble specimens were observed under the Scanning Electron Microscope, as illustrated in Figure 1.

Grain free surfaces of both unpolished and polished specimens were assessed through ImageJ software. In the case of unpolished specimens, the dimensions of the grain surfaces vary, with length in the wide range between 3 μm and 103 μm (average length 39 μm), indicating their inhomogeneous distribution on the specimens' surface (i.e., high SSA value). In the case of polished specimens, the corresponding range is narrow, namely the length ranges between 5 μm and 17 μm, leading to a significantly more homogeneous distribution on the specimens' surface (i.e., low SSA value). Figures 2 and 3 illustrate grain free surfaces of unpolished and polished specimens, respectively.



(**a**)



(**b**)

Figure 1. (**a**) Thin section of unpolished surface; (**b**) thin section of polished surface.

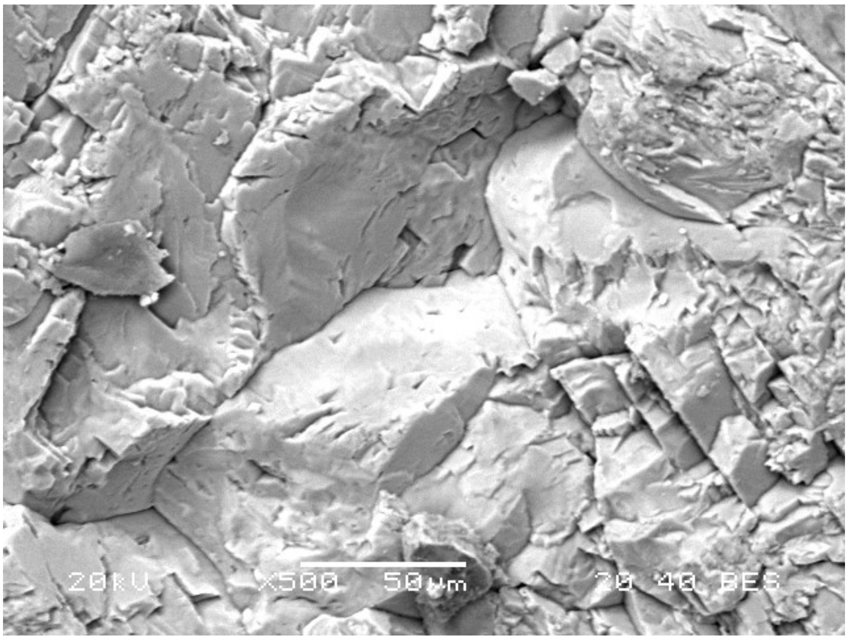


Figure 2. Grain free surfaces of unpolished specimens.

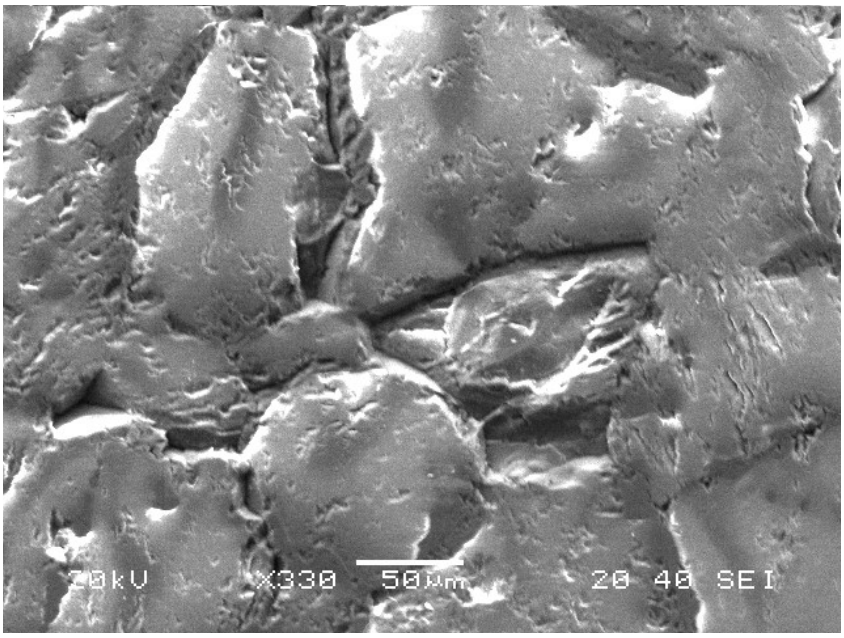


Figure 3. Grain free surfaces of polished specimens.

4. Conclusions

Limestone, granite, calcitic, and dolomitic marble specimens were tested to determine both their slip and frost resistance at LITHOS laboratory according to the applicable EN Standards, EN 14231 and EN 12371, respectively, in order to study the effect of freeze–thaw cycling on their slip resistance.

The Specific Surface Area (SSA), as determined through Equation (1) gives a measure of surface roughness of the specimens. The greater the SSA value of the specimen, the less slippery its surface.

The effect of freeze–thaw cycling is more pronounced on the roughness of unpolished surfaces, mainly under dry conditions, due to the greater surface area exposed to wear.

In general, polished specimens presented almost insignificant change in the measured SRV before and after the exposure in freeze–thaw testing, both under wet and dry conditions, probably due to the observed homogeneity, which seems to prevent slip resistance from being significantly affected. Under wet conditions, the main factor affecting slipperiness of surfaces is water action, which outweighs considerably that of freeze–thaw cycling.

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Informed Consent Statement: Not applicable.

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