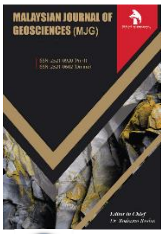
ZIBELINE INTERNATIONAL™ PUBLISHING

ISSN: 2521-0920 (Print)
ISSN: 2521-0602 (Online)
CODEN: MJGAAN

Malaysian Journal of Geosciences (MJG)

DOI: http://doi.org/10.26480/mjg.01.2025.18.27



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RESEARCH ARTICLE

GEOCHEMISTRY, MICROSCRUCTURE AND PHYSICAL PROPERTIES OF THERMALLY STRESSED METAMORPHIC ROCKS

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ARTICLE DETAILS

Article History:

Received 15 February 2025
Revised 19 February 2025
Accepted 25 March 2025
Available online 07 April 2025

ABSTRACT

Metamorphic rocks are useful in many heat-based geotechnical facilities since they are believed to be strong and durable due to their crystalline nature. However, rocks get degraded under repeated thermal stress. This work evaluates the effect of repeated heating and cooling on the geochemistry, mineralogy, microstructure and physical properties of some metamorphic rocks from the Nigerian Basement Complex. Effect of cooling condition was also investigated. Marble, granite gneiss and banded gneiss were subjected to fifty cycles of heating and cooling in air and water using heating temperature between 100 °C and 700 °C. Marble samples used in the study completely disintegrates at the 35th cycle of heating and air-cooling at 500 °C. They could not withstand more than 26 cycles of heating and air-cooling at higher temperature of 700 °C. The porosity, water absorption and degradation degree are highest in marble and least in granite gneiss. The crushing strength of both samples of granite gneiss is higher than that of banded gneiss and marble. Similar trend was observed in the residual values of these physical properties after fifty thermal cycles. Microstructural images showed micro-cracks in the thermally stressed rocks. However, no changes in mineralogy and geochemistry were recorded in the rocks. The water - cooled samples exhibited greater degradation than the air-cooled ones. Granite gneiss is more resilient than banded gneiss while both gneisses are more durable than marble. Metamorphic grade appears to play a major role in the resilience of the rocks to degradation.

KEYWORDS

Crushing strength, degree of degradation, porosity, water absorption, mineralogy

1. INTRODUCTION

Understanding the behaviour of rocks under thermal cycling is essential because it has important applications in geothermal energy development, thermal energy storage systems (TES), climate change resilient infrastructures and deep nuclear waste repositories (Fränzer et al., 2023; Loveridge et al., 2019; Mccartney et al., 2018; Pei and Su, 2023; Wu et al., 2019; Alva et al., 2018; Dincer and Rosen, 2021; Knobloch et al., 2022; Sadeghi, 2022; Sarbu, 2018; Tiskatine et al., 2017, 2016; Costa et al., 2023; Reddy et al., 2024; Barton and Makurat, 2006; Nick Barton, 2020; Prando et al., 2020; Tran et al., 2023).

One of the most pressing global challenge is maintaining necessary energy supplies while mitigating and reducing climate change consequences of burning fossil fuels (Jardine, 2020). This has led to increased quest for new alternative green and sustainable energy sources such as geothermal energy and solar power plants. Geothermal energy is versatile and capable of generating nearly inexhaustible power to meet this demand (Johnston et al., 2011; Pei and Su, 2023; Sánchez et al., 2017). One variant of geothermal resources is the Hot Dry Rock (HDR) which is a high-temperature rock mass that is generally buried 3–10km below the ground surface in the absence of water or steam at temperatures typically ranging from 150°C to 650°C (Hu et al., 2021; Rong et al., 2018; Valencia-González et al., 2015; Yang et al., 2021). Although, geothermal energy have applications in industrial processes, space heating/cooling and electricity generation, the most relevant and much needed use in most developing countries especially Nigeria is electricity generation (Aliyu et al., 2017; Barbier, 2002). During the extraction process of HDR, rocks are exposed to multiple thermal cycling (Hu et al., 2021).

Solar thermal power plants provide sustainable technology for electricity production and it's becoming more popular. It involves the use concentrated sunlight heat to generate steam which drives and produce electricity from turbines. The problem of intermittency associated with this technology can be solved by thermal energy storage (TES) which is the use of rocks as materials for thermal energy storage. It offers an economic and green solution to the problem. Temperatures associated with TES ranges from 150°C to 650°C and it is well known that the bedrock in a thermal energy storage system generally experiences different heating and cooling cycles (Gautam et al., 2021; Rong et al., 2018; Tiskatine et al., 2016; Wu et al., 2019).

Nuclear energy produces enormous energy required to meet the worlds growing need for energy as well as being a green and sustainable alternative to fossil fuels (Brook et al., 2014; Maradin, 2021; Mathew, 2022; Zhan et al., 2021). However, the need for repositories to handle the long - term radioactive waste generated by the energy is immense. Deep geologic repository (DGR) is a versatile long -term storage and its widely agreed to be the best solution for final disposal of most radioactive waste produced (Kurniawan et al., 2022; Ojovan and Steinmetz, 2022). Crystalline rocks are the best-suited rock type for nuclear waste

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repository (Gautam et al., 2021; Laverov et al., 2011). Temperature of rocks used as high level radioactive waste repository can be as high as 900 ° C (Gibb, 1999). A deeper understanding of the thermomechanical stability of rocks plays a crucial role in the selection of suitable host rock for this purpose.

Medium to high thermal cycling can influence the stability and structural integrity of rocks, leading to potential hazards such as landslides and rockfalls. Thermal stress weathering can also deteriorate rocks used as dimension stones through cyclic temperature changes. There is therefore need to incorporate resilience into the design and construction of geotechnical infrastructures, most of which are constructed on and with naturally occurring rocks and soils (Reddy et al., 2024). By studying the resilience of geomaterials under cyclic thermal stress, we can better predict and mitigate the risks associated with changing climatic conditions, contributing to improved infrastructure resilience and public safety.

The effect of thermal cycling temperature and its cumulative effect on the geology and physical properties of rocks in geothermal fields and high-level geothermal waste repository is not common. Previous research has shown that thermal stress due to temperature cycling creates micro pores and variations in flow capacity of HDR and can lead to changes in their physical and mechanical properties (Feng et al., 2020; Kim et al., 2013; Peng et al., 2016; Zhang et al., 2021; Zhu et al., 2019, 2018). However, scanty work has been found on the threshold capacity of rocks under cyclic thermal stress. It is therefore imperative to understand the influence of multiple heating cooling cycles on rocks that are potential HDR(s) and high-level geothermal waste repositories.

About 50% of the Nigerian land mass is underlain by crystalline rocks of various composition and structures (Tijani, 2023). No previous research has been found on the effect of high temperature thermal cycling on the geology and physical properties of these rocks. Rocks are made up of minerals which can be degraded and changed by high temperature and thermal cycling (Li et al., 2021). This work investigates the effect of thermal cycling at different temperatures on three metamorphic rocks from Nigeria. In this work, the effect of prolonged cyclic thermal stress at different intensities on the stability of some Nigerian metamorphic rocks was explored. It is expected provide information on the resilience of these rocks and their potential for use as high-level waste repository, HDR and energy storage rocks.

2. Materials and Methods

Three prominent types of metamorphic rocks namely granite gneisses (GG), banded gneisses (BD) and marble (MB) found in the Nigerian Southwestern basement complex were used for this study (Figure 1).

All the metamorphic rock samples except the dolomitic marble samples were taken from their individual outcrops along Shao-Olooru, SW, Nigeria. The dolomitic marble samples were taken from a quarry site in Oreke, SW, Nigeria. Each sample taken was inspected to ensure that it was fresh and free of macroscopic defects so that it would provide test specimens free from fractures.

The fresh rock samples were subjected to basic tests such as water absorption, porosity and crushing strength tests. Their mineralogy geochemistry, microstructure and geochemistry were also investigated. The rock samples were subjected to cyclic heating and cooling up to fifty cycles at predetermined temperatures of 100 ° C, 300 ° C, 500 ° C and 700 ° C. Each cycle of thermal stress involves heating the rocks in an electric oven for two hours, and air/water cooling them. The rock samples were heated to the desired temperature at the rate of 20 ° C/ min in the oven and cooled in water bath (for water cooling) or desiccator (for air cooling). Only samples heated to 700 ° C were water cooled in water bath after heating, the rest were cooling using desiccator. This was done to investigate the role water plays in observed changes in the rock due to thermal cycling. The effects of this cyclic thermal stress (heating and cooling) on their mineralogy, geochemistry, microstructure and some mechanical properties of the rocks such as porosity, water absorption and crushing strength were investigated.

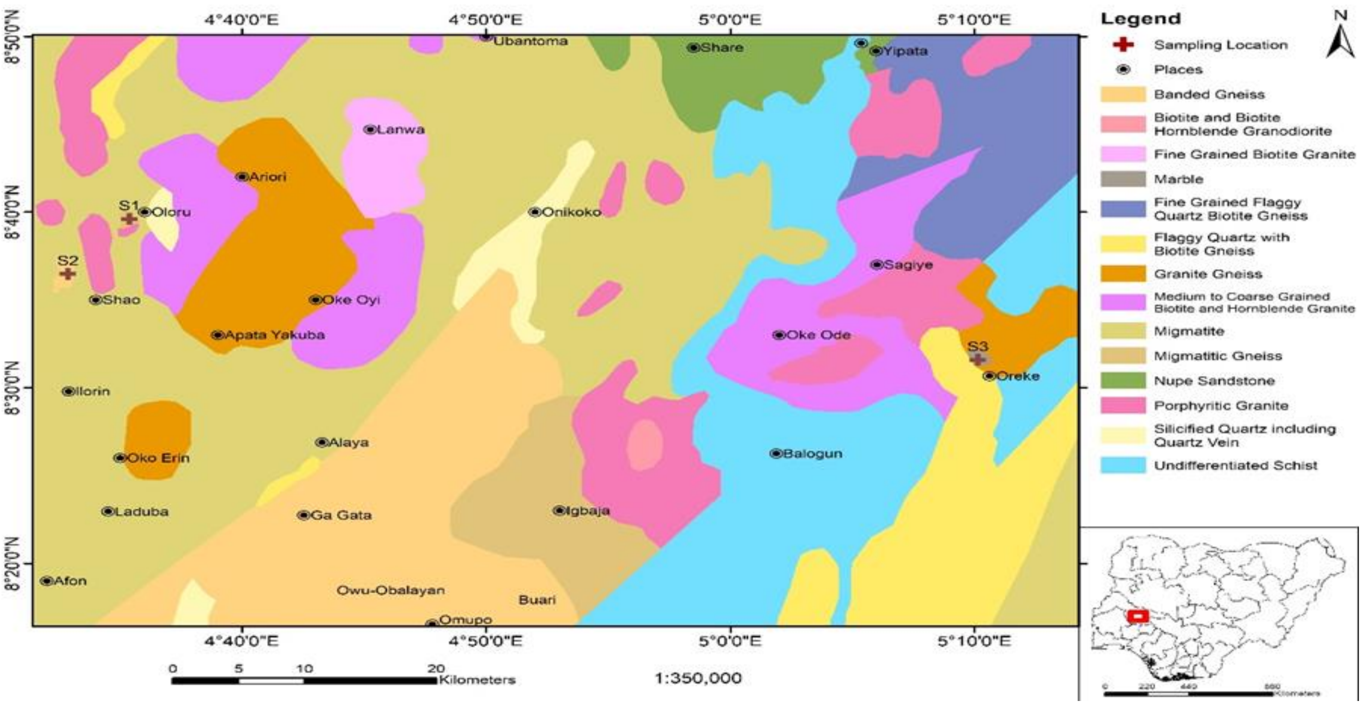

Figure 1: Geologic map showing locations of the sampling sites

X-ray diffractometry (XRD) was used to analyse the minerals in the soil before and after thermal cycling using a diffractometer. Evaluation of the diffractograms was done using the database of the International Centre for Diffraction Data (ICDD PDF -2®). Their geochemistry was determined using and X-ray fluorescence (XRF) spectroscopy. Major oxides excluding sodium present in each rock was determined by fusion disc prepared from 0.28g of each rock sample and a mixture of lanthanum oxide, lithium borate, lithium carbonate and sodium nitrate. Sodium was measured using pressed pellet prepared from a mixture of 8g rock flour and 3g Hoechst wax. Total loss on ignition (LOI) was determined by heating 10g each of pulverized rocks to 110°C. The microstructure of the rocks was studied using a scanning electron microscope equipped with an energy dispersive spectroscopy detector.

The porosity of the rock samples was determined by the saturation method. The rock samples were saturated by water immersion for at least 24 h. After sample saturation, the surface of the samples was dried with a moist cloth and the saturated surface dry weight was measured. The saturated samples were oven dried (at 105°C) for 24 h after which the weight of the oven-dried samples was determined. The difference between the saturated surface dry weight and the dry sample weight gave the effective pore volume, which was used to calculate the porosity. 75cm by 75cm cubes (Figure 2) were prepared from each boulder for uniaxial compressive strength test (crushing strength) following ASTM standard test methods C170 (ASTM, 2016). Care was taken to ensure uniformity that all cubes and other irregular samples used for all analysis were taken from same boulder. In determining the UCS of the rocks, cubic samples were loaded on the base plate of universal testing machine. The samples were loaded until failure. The load at failure (F) was recorded and crushing strength (C_o) was calculated using (1).

$$C_o = F/A \quad (1)$$

Where A is cross sectional area, C_o is Uniaxial compressive strength, F is the Total load at failure, A is cross sectional area.

Changes in the water absorption, porosity, crushing strength, geochemistry, microstructure and mineralogy of the rock samples were investigated. The degradation degree of a certain physical and mechanical parameter is characterized by its relative change, that is, the ratio of the difference between the parameter value of the rock sample after the dry-wet cycle and the initial value to the initial value (Zhang et al., 2021). The degradation of the rocks from each rock property were calculated using equation 2.

Figure 2: Rock samples used for unconfined compression test.

$$G_n = \frac{P_n - P_O}{P_n} \times 100 \quad (2)$$

Where G_n is the degradation degree at thermal cycle n, P_o is parameter value of fresh rock sample, and P_n is the value of parameter after n number of thermal cycle.

3.Results and Discussion

3.1 Porosity

Several factors influence rock porosity, including grain size, grain shape, and the arrangement of rock grains, as well as the degree of deformation and geological processes such as mineralogical and geochemical alterations (Peng & Zhang, 2007). In this study, the natural porosity of unheated rock samples follows the order: MB > BG > GG. As temperature cycling increased, the porosity of all rock samples rose proportionally (Figure 3). The degree of degradation also increased with higher thermal cycling temperatures, as reflected in the porosity changes. Table 1 presents the degradation degree and percentage change in porosity of the rock samples after 50 cycles of thermal stress. It is noteworthy that while both BG and GG exhibited a significant increase in porosity at 700°C, MB could not withstand thermal stress beyond 300°C. MB disintegrated completely by the 35th cycle when subjected to a thermal stress temperature (TST) of 500°C. In contrast, both BG and GG, which had lower initial natural porosity, demonstrated greater resilience, withstanding larger porosity changes without disintegrating. Although GG showed more resistance to porosity increase than BG across all temperature ranges, MB remained relatively stable at lower TSTs, particularly at 100°C (see Figure 3).

When the thermally stressed rocks were cooled with water, a greater degree of degradation was observed, as reflected in the increased porosity. This suggests that water accelerates degradation by promoting more rapid increases in porosity. Initial porosity appears to be the primary factor driving these changes in thermally stressed rocks. MB, composed predominantly of recrystallized dolomite and calcite (see Table 4), is structurally weaker compared to BG and GG, which are silica rich. Furthermore, the alternating leucocratic and melanocratic mineral bands in BG contribute to its relatively high porosity by introducing structural weaknesses along these bands.

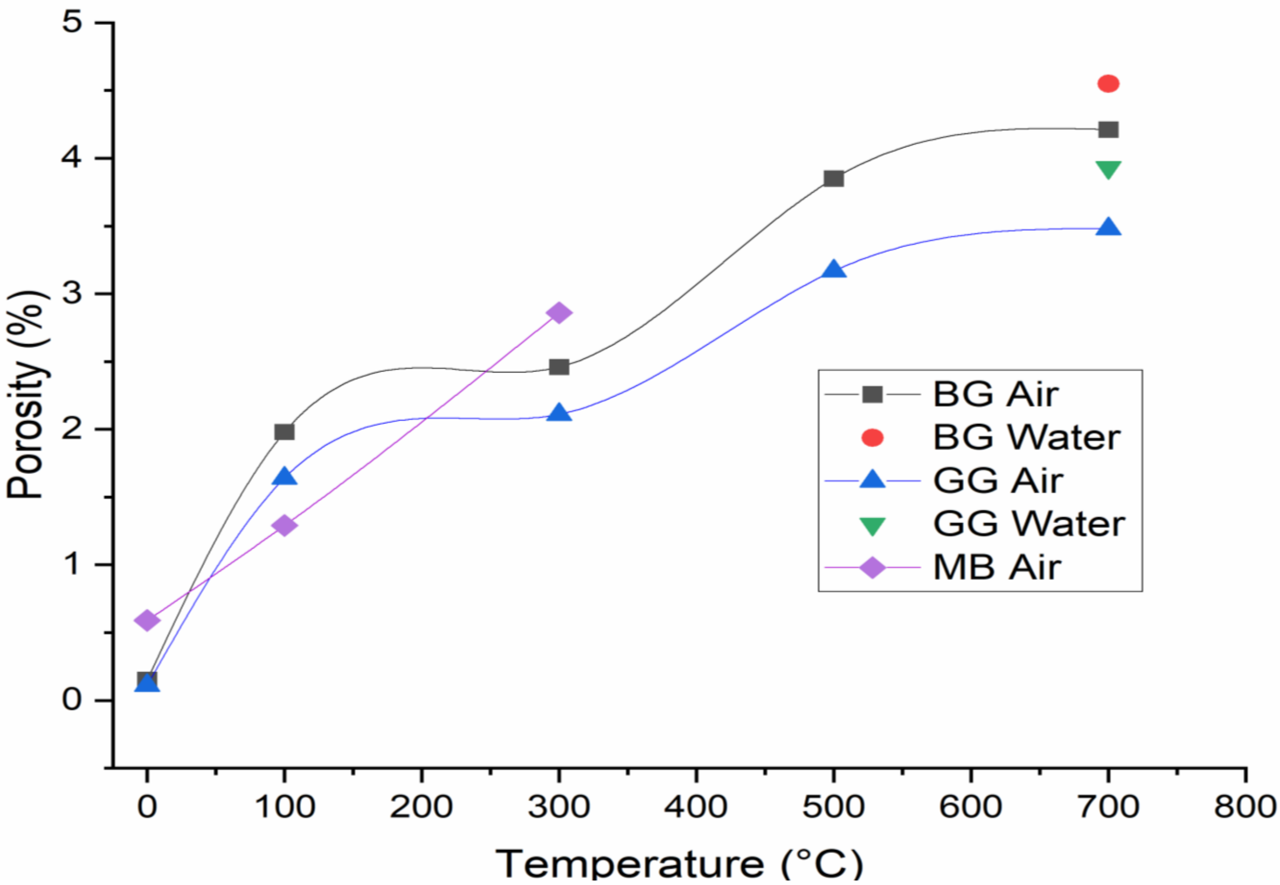

Figure 3: Variation of porosity with temperature of thermal cycle

Table 1: Porosity and degradation degree using porosity as yardstick.			
Porosity (%)	**Banded gneiss (BG)**	**Granite gneiss (GG)**	**Marble (MB)**
Fresh rock	0.15	0.11	0.59
R_{100}	1.98	1.64	1.29
R_{300}	2.46	2.11	2.86
R_{500}	3.85	3.17	-
R_{700air}	4.21	3.48	-
$R_{700water}$	4.55	3.93	-
Temperature of thermal cycles	G_{50} (%)	G_{50} (%)	G_{50} (%)
100	1220	1391	119
300	1540	1818	385
500	2467	2782	-
700air	2707	3064	-
700water	2933	3473	-

*R (Residual value of parameter after 50 thermal cycle), G_{50} (Degradation degree after 50 thermal cycle)

3.2 Water absorption

The ability of rocks to absorb water plays a critical role in their structural integrity and containment capacities (Guiling et al., 2017; Zhang et al., 2022). Rocks with appropriate water absorption capacity can enhance thermal conductivity and improve energy transfer mechanism in geothermal systems. Figure 4. presents the variation in water absorption of the rock samples after 50 cycles of heating and cooling at different temperatures. For all rock types, water absorption increased with temperature of thermal cycles.

However, MB showed a limitation, being unable to withstand thermal cycle temperature beyond 300°C A similar trend to the one observed in porosity is also evident here: water absorption is higher in MB compared to BG and GG. This relationship is presented in Table 2, which shows the degradation degree, or percentage change in water absorption capacity, due to cyclic thermal stress at varying temperatures.

An increase in porosity directly correlated with greater capacity for water absorption in the rocks. The samples that were cooled in water exhibited higher water absorption and degradation degree higher than those cooled in air. This can be attributed to rapid cooling induced by water, which creates more extreme temperature fluctuations and accelerates the degradation process. Water cooling forces the rock to cool at a much faster rate, intensifying the thermal shock, and leading to more substantial structural breakdowns.

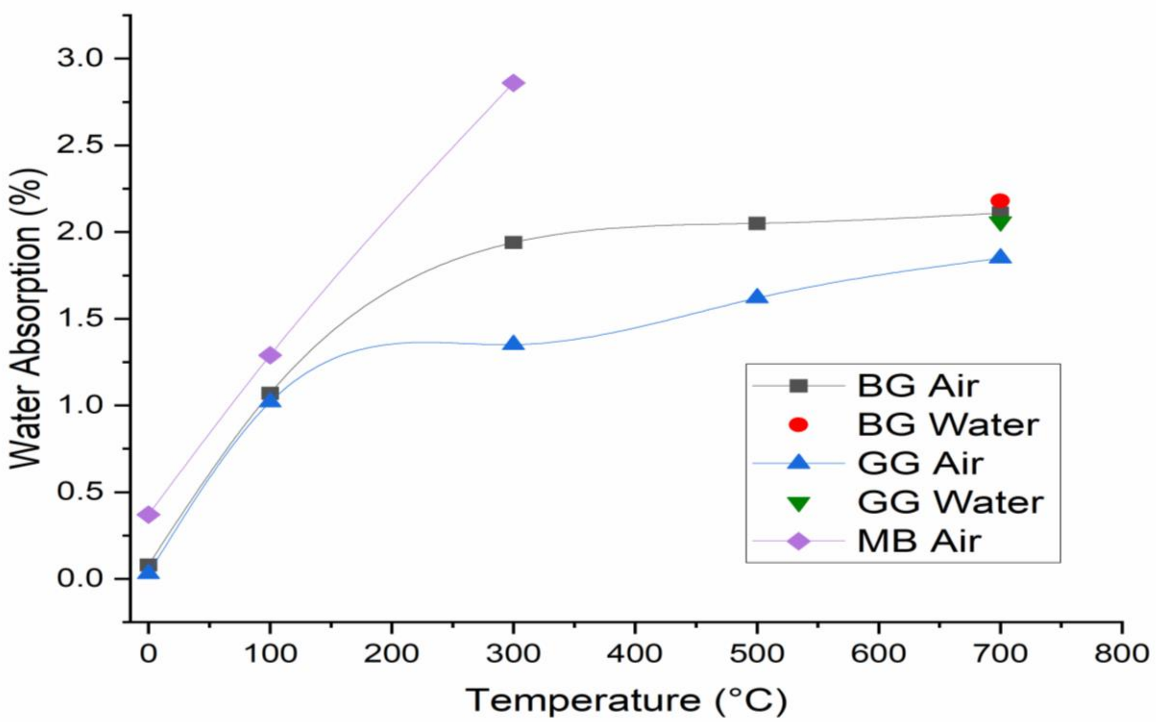

Figure 4: Variation of water absorption with cycling temperatures of the rock samples.

Table 2: Water absorption capacity and degradation degree using water absorption as a determinant			
Water absorption capacity (%)	**Banded gneiss**	**Granite gneiss**	**Marble**
Fresh rock	0.08	0.03	0.37
R_{100}	1.07	1.02	1.29
R_{300}	1.94	1.35	2.86
R_{500}	2.05	1.62	-
R_{700air}	2.11	1.85	-
$R_{700water}$	2.18	2.06	-
Temperature of thermal cycles	G_{50} (%)	G_{50} (%)	G_{50} (%)
100	1238	3300	249
300	6200	4400	673
500	6567	5300	-
700air	6767	6067	-
700water	7000	6767	-

3.3 Crushing strength

Unconfined compressive strength (UCS) is a key indicator of the crushing strength of rocks. Figure 5 illustrates the effects of thermal cycling on the crushing strength of the metamorphic rock samples. The data suggest that the initial (or fresh) strength of each rock largely determines the residual strength after repeated heating cycles across all temperatures. Granite gneiss (GG) exhibited the highest initial strength, while marble (MB) had the lowest, with banded gneiss (BG) falling in between.

Residual strength consistently decreased with increasing temperature across all samples. MB completely disintegrated after 35 cycles at a TST of 500°C, while at 700°C, this threshold dropped to 26 cycles (see Figure 5). Table 3 further highlights that GG maintained the highest residual crushing strength at all temperatures, whereas MB exhibited the lowest. The degradation degree, measured in terms of crushing strength loss, was highest in MB and lowest in GG, indicating that GG is the most resistant to strength degradation. In contrast, BG was more resistant than MB but less resistant to degradation than GG.

The varying degrees of strength degradation can be attributed to the mineralogical and textural differences between the rocks. GG and BG are predominantly composed of quartz and feldspars, minerals known for their strength, while MB is primarily composed of calcite and dolomite, which are relatively weaker minerals. The geological processes responsible for forming these rocks also play a significant role. GG and BG were subjected to high-grade metamorphism—conditions of elevated temperature and pressure—which resulted in dense grain packing and strong interlocking textures. This structural integrity enhanced resistance to crushing, particularly in GG. On the other hand, MB, which formed under intermediate to high-grade metamorphic conditions, lacks the same degree of interlocking and dense grain structure, making it more susceptible to crushing strength degradation. The lower initial and residual crushing strength in BG can also be explained by its pronounced foliation, a structural feature that weakens the rock by introducing planes of weakness. While BG possesses interlocking grains, the foliation reduces its overall resistance to crushing, especially after repeated thermal cycling (see Figure 2).

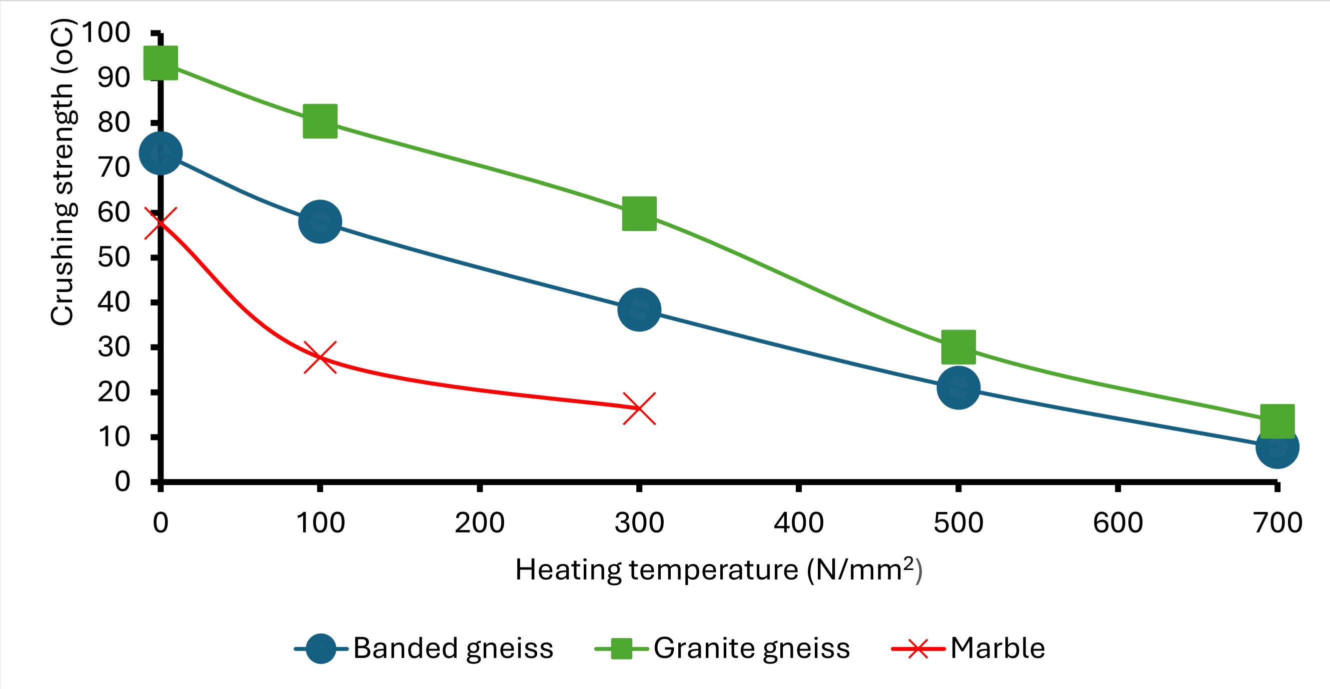

Figure 5: variation in crushing strength of the rock's temperature of thermal cycle

Table 3: The crushing strength and degradation degree using crushing strength as yardstick.			
Crushing strength (%)	**Banded gneiss**	**Granite gneiss**	**Marble**
Fresh Rock	73.24	93.33	57.6
R_{100}	57.96	80.36	27.73
R_{300}	38.4	59.73	16.36
R_{500}	20.98	30.04	35th cycle*
R_{700air}	10.31	19.56	26th cycle*
$R_{700water}$	7.82	13.51	23rd cycle*
Temperature of thermal cycles	G_{50} (%)	G_{50} (%)	G_{50} (%)
100	20.9	13.9	51.9
300	47.6	36.0	71.6
500	71.4	67.8	-
700air	85.9	79.0	-
700water	89.3	85.5	-

*Cycle: Complete disintegration of sample occurred at designated cycles before the 50^{th} cycle was completed.

3.3.1 Effect of thermal cycling on the mineralogy of the rocks

The mineralogical composition of the rock samples after undergoing fifty cycles of alternating heating and cooling, either in water or air at 700°C, is shown in Table 4. The X-ray diffractograms of these thermally stressed rock samples are presented in Figure 6. Overall, no significant changes in the mineral composition were observed in most of the rock types. However, an exception was noted in the banded gneiss (BG) samples, where the presence of amphiboles appeared, and an increase in K-feldspar content was recorded, particularly in the air-cooled specimens.

The mineralogical alterations in BG may be attributed to the inherent inhomogeneity within the rock, rather than genuine chemical changes induced by thermal cycling. This suggests that, for the most part, no substantial chemical transformations occurred in the rock samples. Instead, the primary impact of thermal cycling seems to be physical degradation, driven by the repeated expansion and contraction of mineral grains. This physical breakdown, rather than chemical alteration, is likely the result of thermal stress from the cyclical heating and cooling process, which causes the minerals to expand when heated and contract upon cooling. In BG, the increase in K-feldspar could indicate a redistribution of mineral phases within the rock matrix due to thermal cycling, while the appearance of amphiboles might reflect local compositional variations.

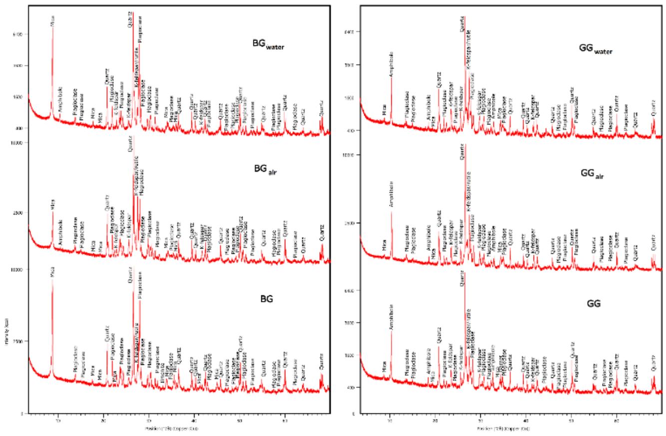

Figure 6: X-ray diffractograms of fresh samples BG and GG and their thermally degraded counterpa

3.4 Effect of thermal cycling on the geochemistry and microstructure of the rock samples

No significant changes in the chemical composition of the thermally stressed samples (Table 4). This lack of chemical alteration helps explain the minimal mineralogical changes detected in the samples, with only physical breakdown occurring. In addition, the absence of geochemical changes suggests that the thermal cycling primarily induced mechanical degradation, rather than any substantial chemical transformations within the rocks. Figure 7 presents the scanning electron micrographs (SEMs) of fresh samples BG and GG samples alongside those that underwent thermal cycling at 700°C, followed by cooling either in air (BG_{air} and Ga_{it}) or in water (BG_{water} and GG_{water}). The micrographs reveal a series of micro-cracks and micro-pores in the thermally stressed samples, with the water – cooled rocks showing more pronounced damage compared to the air - cooled ones. This pattern aligns with previous findings by Zhang et al. (2021),Qin et al., (2024) and Wang et al., (2024) who reported that water cooling tends to generate more severe thermal shock, leading to an increased formation of micro-cracks and pores. The formation of these micro-cracks and pores is likely due to the differential thermal expansion and contraction of mineral grains within the rocks. Water cooling allows for more abrupt temperature drops, which intensifies the mechanical stress on the rock structure. In contrast, air cooling, although still contributing to degradation, results in slightly less damage due to the more rapid yet less intense thermal gradient.

Table 4: Effect of thermal cycling on the geochemistry of the rock samples

Weight (%)	MB_{fresh}	MB_{air26}	$MB_{Water23}$	GG_{fresh}	GG_{air}	GG_{water}	BG_{fresh}	BG_{air}	BG_{water}
SiO_2	4.22	5.63	2.93	65.60	66.30	66.58	64.43	67.50	63.87
TiO_2	0.00	0.00	0.00	0.68	0.55	0.61	1.06	0.61	1.26
Al_2O_3	0.00	0.00	0.00	11.88	12.58	12.87	14.45	14.62	14.94
Fe_2O_3	0.06	0.08	0.08	10.03	7.89	9.02	6.59	4.29	7.18
MgO	21.74	21.69	21.66	0.10	0.27	0.13	1.56	0.89	1.54
MnO	0.00	0.05	0.00	0.14	0.10	0.12	0.08	0.05	0.09
CaO	30.99	31.27	31.59	3.27	2.76	3.08	3.57	2.50	3.75
Na_2O	0.06	0.01	0.01	1.40	1.52	1.54	3.28	2.47	2.29
K_2O	0.00	0.00	0.00	4.81	5.86	5.66	2.85	4.88	3.33
P_2O_5	0.01	0.01	0.01	0.13	0.11	0.11	0.47	0.29	0.59
LOI	41.86	41.03	41.79	0.07	0.10	0.15	0.22	0.20	0.29
Total	98.95	99.72	98.06	98.10	98.04	99.86	98.55	98.31	99.13

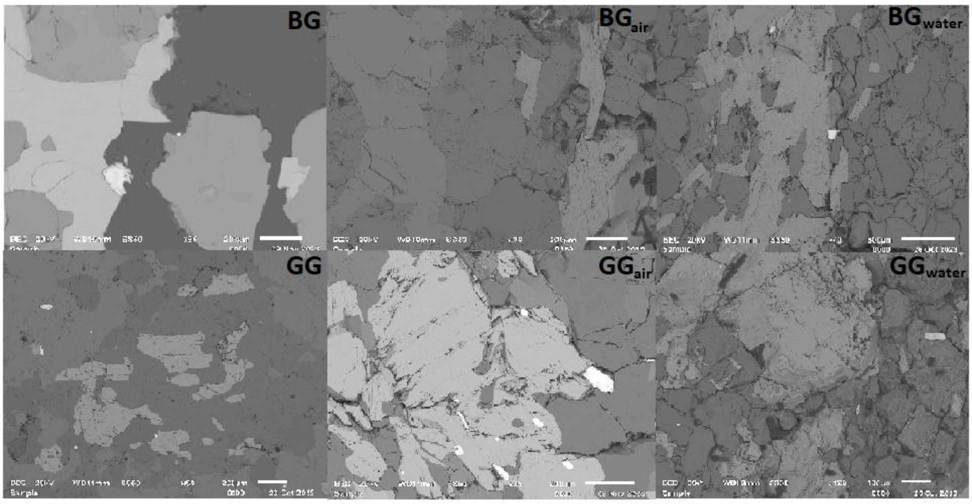

Figure 7: Microstructural images of the fresh and thermally stressed samples.

3.4 Effect of cooling condition on rock properties

As shown in Figure 8, after the 50 thermal cycle, the residual crushing strength of the air - cooled f BG and GG samples is greater than that of the water – cooled ones. Likewise, residual porosity and water absorption capacity of the air - cooled rock samples are lower than those cooled in water. This implies that presence of water expedites degradation in thermally stressed rocks. Water cooling subjects the samples to more rapid cooling, causing faster contraction and expansion of the mineral grains. This intensifies the thermal stress, leading to more pronounced mechanical breakdown compared to air-cooled samples. Due to the marble (MB) samples' inability to withstand 50 thermal cycles at 700°C, they were excluded from direct comparison. The marble samples completely disinterated at the 26th cycles when air-cooled and after 23rd cycles when water-cooled at 700°C. This further illustrates the impact of cooling conditions, as the air-cooled MB samples withstood three more cycles than those cooled in water. The faster cooling rate in water exacerbates thermal shock, leading to quicker failure.

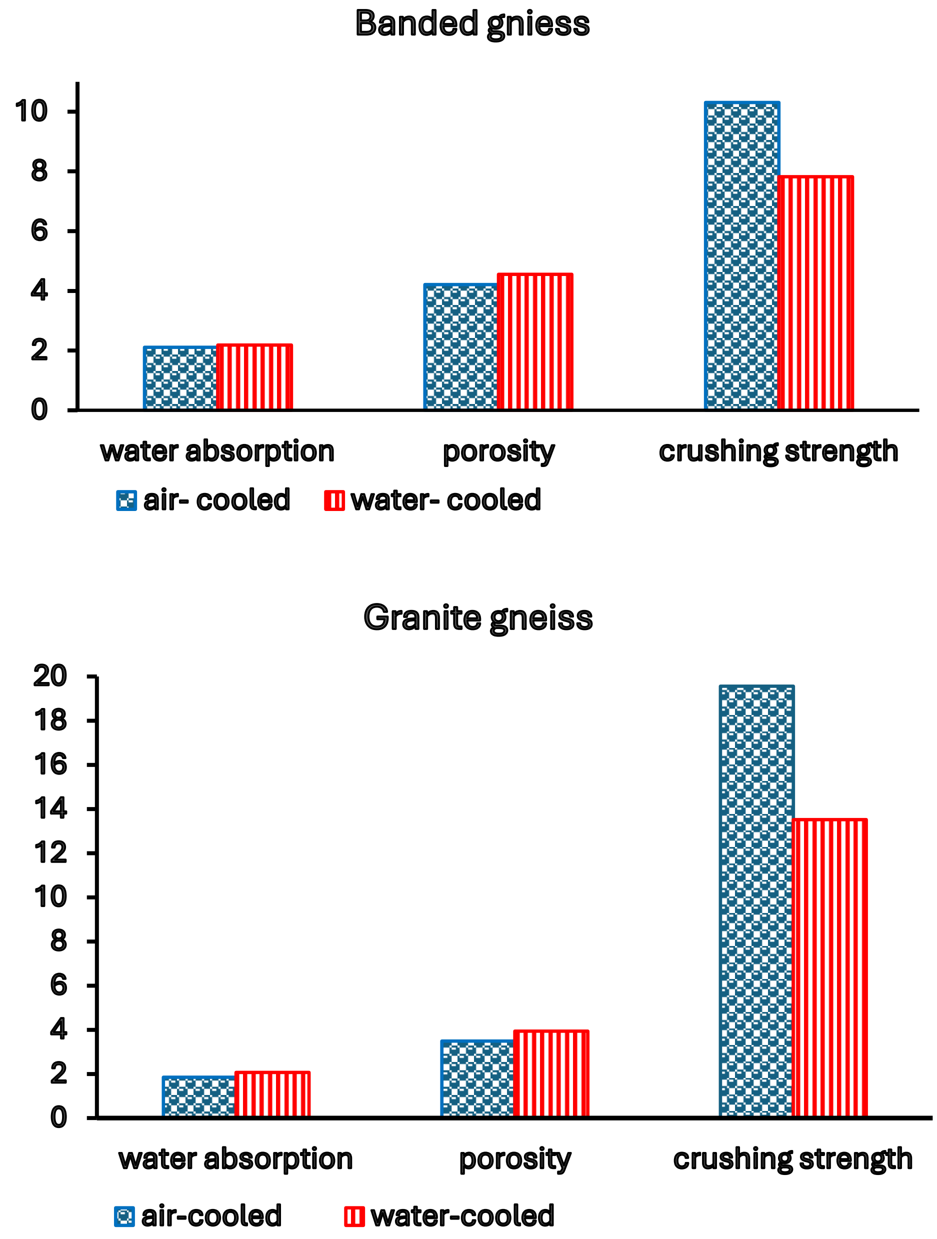

Figure 8: Effect of cooling condictions on the physical condiction of the rocks after 50 cycles of alternate heating and cooling

4. Conclusion

Samples of three metamorphic rocks—marble (MB), banded gneiss (BG), and granite gneiss (GG)—were subjected to alternating heating and cooling cycles at temperatures ranging from 100°C to 700°C, up to fifty cycles. The effect of thermal stress on the porosity, water absorption, crushing strength, mineralogy, geochemistry and microstructure of the rocks were investigated, along with the effect of cooling conditions (either in air or water).

- Fresh samples of marble exhibited the least strength, the highest porosity and water absorption compared to other rocks. Marble completely disintegrated at the 23rd cycle when water-cooled and after 26th cycle when air-cooled, under TST of 700 °C. This demonstrates marble's low resistance to thermal stress, particularly under water-cooled conditions, which expedited its degradation. No significant changes in the mineralogy or geochemical composition of any of the rocks were recorded after the thermal cycles. This suggests that the primary effect of thermal stress was physical rather than chemical, as the rocks maintained their mineral compositions throughout the experiment.

- Scanning electron microscopy images revealed the formation of micro–cracks and micro–pores in the thermally stressed samples. These features were more pronounced in water-cooled samples, highlighting the role of rapid cooling in inducing microstructural damage.

- The presence of moisture during cooling process (water-cooled samples) significantly exacerbated the degradation of the rocks.

Water-cooling resulted in more rapid contraction, intensifying the effects of thermal stress and leading to more severe mechanical breakdown compared to air-cooled samples.

- The degree of degradation under cyclic thermal stress was strongly influenced by the rock's grade of metamorphism and mineral composition. Rocks with higher-grade metamorphism and mineral content dominated by quartz and feldspar (such as GG and BG) were more resistant to thermal cycling than marble, which consists mainly of calcite and dolomite. The denser grain packing and stronger interlocking of mineral grains in higher-grade metamorphic rocks provide better resistance to thermal stress.

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