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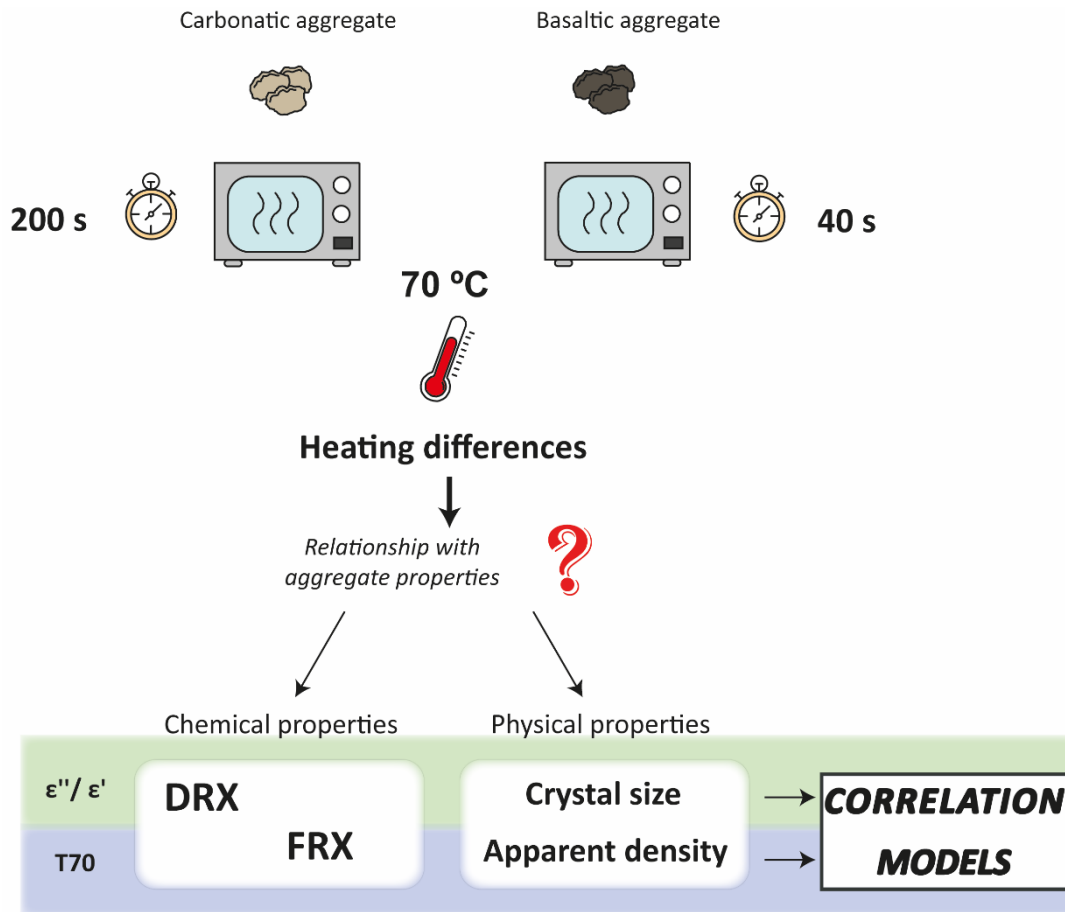
Natural factors related to the differential heating of aggregates exposed to microwaves

Keywords

Aggregates. Microwave heating. Petrography. Mineralogy. Chemical composition. Asphalt mixtures. Self-healing.

Highlights

- The chemical and mineralogical composition of aggregates affects microwave heating.
- Models have been proposed to predict microwave heating of aggregates for asphalt pavements.
- A relationship has been established between crystal size and heating in mono-mineral rocks.



Abstract

The heating produced by microwaves has been demonstrated to be effective in materials such as aggregates, although notable differences in heating have been observed between different types of aggregates. The geological factors of each material are responsible for these differences in heating due to compositional elements such as the mineralogy and chemistry of the material and physical characteristics such as crystal size and apparent density.

The study of each of these characteristics could unravel the **factors** determining differential heating in aggregates, which would be relevant for different applications in the mining industry (heating, drying, and comminution) and in civil engineering in self-healing (microcrack repair) or winter road maintenance (surface de-icing).

This paper investigates the influence **of** microwave heating **on** the chemical, mineralogical, and physical properties of aggregates susceptible to be used in asphalt mixtures. For this, aggregates with different characteristics have been selected. Various

1 tests were conducted to determine the factors affecting heating: thin-section studies
2 (mineralogy and crystal size), XRD (mineralogy) and XRF (chemistry) composition
3 studies, and bulk density of the rocks. These results were correlated with dielectric
4 permittivity data (loss tangent) and direct heating data (parameter T70). Both
5 techniques have proven helpful as an indicator of susceptibility to microwaves. Finally,
6 multiple regression models were proposed to apply this knowledge to any rocks suitable
7 as aggregate.
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13 14 15 16 **1. Introduction**

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18 Aggregates are essential materials for society and are in high demand [1] for
19 different applications such as the manufacture of concrete and mortars, aggregates for
20 pavements, breakwaters, ballast, and other applications [2]. The sources of aggregates
21 are varied; for example, in Spain, the quarries of crushed stone stand out with 69.8%,
22 followed by sand and gravel pits with 25.7%, although there are by-products from other
23 mining operations 2.7%, and a growing but still low (1.9%) use of recycled and artificial
24 materials [2], which in some cases are also pre-treated to improve their properties [3,4].
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33 One of the highest aggregates consumption activities is road construction, using
34 approximately 28% of the aggregates. In addition to aggregates, asphalt mixtures
35 include other materials such as bituminous compounds, a primary source of carbon
36 emissions [5]. The current interest in climate change and the reduction of carbon
37 emissions have motivated recent research on the preventive maintenance of
38 pavements. One promising method is heating asphalt pavements by microwaves until
39 the bituminous material flows, producing the healing of cracks in a process known as
40 self-healing [6,7]. The studies carried out in the laboratory confirmed this technique as
41 an effective tool to extend the life span of asphalt pavements.
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51 Microwave heating is a green technology that demonstrates many advantages
52 over conventional heating methods. Microwave energy is rapidly transformed into heat
53 within the material through electromagnetic radiation. In contrast, in conventional
54 heating, thermal energy has to cross through the volume of the sample from the outside
55 [8]. Microwave heating could be considered a clean process since it uses electrical
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energy compared to other processes where heat is generated through combustion [9]. The advantages of this type of heating are rapid and contactless heating, selective heating of the material, volumetric heating (from inside of the material), and a high level of safety for operators [10–12], besides environmental advantages such as the reduction of atmospheric emissions [13]. Microwave applications in mineral processing have focused on assisted drying and crushing. Still, recently, other applications have emerged, such as mineral and rock heating, for applications related to the road industry, such as material heating to reduce manufacturing costs [13] or self-healing [14].

Microwave frequencies cover a part of the electromagnetic spectrum that extends from 300 MHz to 300 GHz [15]. The dielectric loss tangent defines the ability of a material to heat up in the presence of a microwave field:

$$\operatorname{tg} \delta = \frac{\varepsilon''}{\varepsilon'} \quad (1)$$

The dielectric loss tangent comprises two parameters, the dielectric constant (ε') and the dielectric loss factor (ε''). The dielectric constant or real permittivity determines the amount of incident energy not absorbed by the material. In contrast, the dielectric loss factor or imaginary permittivity measures the dissipation of electrical energy inside the material in the form of heat [10–12].

The factors that could influence heating, in addition to the real and imaginary permittivity (that depend on the frequency), are thermal conductivity, heat capacity, temperature, apparent density, sample size, particle size, presence of susceptors, chemical composition, microwave cavity geometry, and power level [15]. In addition, water content is often a contributing factor since water has a high loss factor [16].

Studies on the interaction of microwaves with minerals and rocks are scarce. Some works have attempted to classify rocks and minerals according to their responses to microwave radiation (rate of heating and maximum temperature) at different power levels. Still, the diversity of the studied rocks is very limited. One of the pilot studies related to civil engineering was carried out by Benedetto & Calvi [13], who tested two types of rocks (basalt and limestone), obtaining good heating results for basaltic

aggregates. Another study, which relates heating to mineralogy, is that of Garcia-Baños *et al.* [17], where four types of rock were studied (basalt, granite, sandstone, and marble), obtaining results similar to those of [13]. A study carried out by Li *et al.* [18] classifies rock heating by its origin, obtaining good results with higher heating for igneous rocks with basic composition than heating in igneous rocks of acid composition. However, sedimentary rocks presented a similar behaviour to igneous rocks of acid composition. Finally, the most complete studies in this area are those of Trigos *et al.* [14,19], in which various types of aggregates corresponding to blast furnace slag, carbonate (limestone and dolomite), volcanic (ophite, andesite, and basalt), plutonic (granite and granodiorite), and metamorphic rocks of different grades were studied. The results obtained from heating suggested that the composition of the rocks plays a key role in their differential heating.

Other studies focused on the behaviour of minerals instead of rocks. The first study is Chen *et al.* [20], which measured the heating of 40 minerals belonging to different groups. This work determined that the minerals that present more heating are sulphides and oxides with a metallic appearance; in contrast, minerals of the group of carbonates and silicates have low heating rates. Additional studies on the heating of sulphides obtained similar conclusions [18,21]. Although some studies showed that rock-forming minerals (silicates and carbonates) are weak microwave absorbers (absorbing microwave energy transforming it into heat), they are not completely inert to microwave radiation. The information on these minerals from different authors and their relationship to microwaves is consistent and allows the minerals to be classified according to their microwave absorption capacity. Prevalent minerals considered weak microwave absorbers are quartz, k-feldspar, plagioclase, and calcite [12,13,16,21–26]. Other common minerals, such as muscovite and hornblende, are considered medium absorbers [12,16,21,23]. Finally, biotite and pyroxene (enstatite) are good absorbers [12,23,26,27]. Other less common minerals are olivine and garnet (almandine), which behave as weak microwave absorbers [23]. It should be noted that microwave absorption and response can be conditioned by the origin, type, and content of impurities [12].

1 The influence of the chemical composition in the microwave heating process has
2 been studied in some works. The data collected from different authors allowed us to
3 differentiate three groups: high absorption (Fe_2O_3 , MnO , and TiO_2) [21,23,26,28–33],
4 medium absorption (Al_2O_3 , MgO , and Na_2O) [28,30–33], and low absorption (SiO_2 , K_2O ,
5 and CaO) [28,30,31]. Other compounds that facilitate heating even in trace
6 concentrations, and have high absorption, are NiO_2 , CoO , CuO , WO_3 , and CrO_3 [21,31].
7 The effect of elemental substitution and impurities can impact the permittivity of
8 minerals. Iron, chromium, and manganese oxides have higher permittivities than other
9 oxides [21].
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11 Another factor to consider is the crystal size and rock texture, related to the
12 durability of the rocks [34]. The most relevant work on this topic in microwave heating
13 is Nicco *et al.* [24]. Minerals that efficiently absorb microwaves and have high dielectric
14 constants are known to facilitate heating. However, the spatial distribution of these
15 grains is crucial since the heterogeneity of rock materials is essential in the response of
16 rocks subjected to microwave irradiation [12,35]. Therefore, heterogeneous properties
17 produce non-homogeneous thermal distribution and selective absorption between
18 minerals [24]. Fine-grained rocks can be heated more efficiently due to an adequate
19 distribution between the crystals since the heated minerals are closer [36,37]. On the
20 other hand, in coarse-grained rocks, the redistribution of heat is more inefficient,
21 causing the rock not to heat homogeneously and producing hot-spots [26,27].
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23 Some authors [33,38] have considered the apparent density factor in the
24 differences obtained in the dielectric permittivity. Other physical properties associated
25 with the density are the porosity [39] and water content [21,40] that can affect
26 permittivity and heating. A general classification of the influence of water present in the
27 material informed about its permittivity and microwave response was proposed by [21].
28 This classification is sorted from weakest to strongest: bulk water, surface, weakly
29 adsorbed, strongly adsorbed, hydrated, and hydroxyl.
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31 This study aims to know the chemical or physical properties that influence
32 microwave heating of aggregates that give rise to differential heating for industrial and
33 construction applications.
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Several emerging technologies can benefit from this type of heating regarding the practical application of microwave radiation in asphalt materials and pavements. For example, some of the related microwave applications in the aggregates industry are material drying [41], crushing or industrial heating [42,43]. Other studies, already directly related to asphalt pavements, are for manufacturing asphalt mixtures, replacing the combustion of fossil fuels [13]. Likewise, prototypes have been proposed for the de-icing of the pavement in winter, based on the projection of radiation on the pavement [30]. These types of devices, already existing, allow heating pavement sections of the order of 1 m² to perform localised conservation operations [44] that could be applied on a larger scale, for in situ recycling of asphalt pavements or to promote the assisted repair of the most superficial layers of the pavement [45].

Regarding the repair of pavements, it must be taken into account that the asphalt mixture is made up of a combination of aggregates, additives, and bitumen, the aggregate being the main constituent with more than 90% by weight of the mixture, so the aggregates form an essential part of the asphalt mix. Furthermore, regarding the interaction of each of these components with microwaves, it is known that microwave heating is strongly influenced by the electromagnetic characteristics of the irradiated material, improving the efficiency of the system. The heating of the pavement is produced by the heating of the aggregates and the additives that compose it [7] since bitumen is transparent to microwaves [46]. Therefore, the heating of the asphalt pavement depends mainly on the aggregates to absorb the energy and transfer it to the asphalt.

Therefore, the results of this research may apply to these technologies in the construction and maintenance of roads. Furthermore, it should be considered that the electrical energy used by microwave devices, when it comes from renewable sources, notably improves the sustainability of these tasks, compared to traditional construction and conservation procedures, based on the consumption of fossil fuels. Hence the final objective of this research is the proposal of aggregates that optimise the energy consumption of these emerging technologies due to their response to microwave energy.

In order to know which aggregates have the best susceptibility properties (microwave heating), an experimental study was carried out on a broad spectrum of samples to determine the influence of the chemical and physical characteristics of the rocks in their response to heating in the face of the microwave exposure. A total of 24 samples were studied, some with similar compositions but different textures. A detailed analysis of the chemical properties (mineralogy and chemistry) and the physical properties (crystal size and apparent density) of the rocks under study has been carried out. In addition, microscopic observations were made to determine whether the warming causes are controlled by mineralogy, texture, or grain size. In addition, knowledge is offered on the influence of bulk density and grain size and the content of some chemical compounds such as iron, hydrated minerals, and their relationship between the dielectric properties of the rock.

Material characterisation is important in determining the applicability of microwaves to different rock types and optimising assisted processes by predicting materials that lead to efficient heating processes. The long-term objective is to provide knowledge that can propose rock types for industrial or construction techniques to make them more profitable from an economic and environmental point of view.

2. Materials and Methods

2.1. Materials

Twenty four aggregates of different compositions and origins have been studied (Table 1 and Figure 1). According to their composition, the rocks have been grouped into two categories: **a) Silicate aggregates**, ordered from highest to lowest SiO₂ content. Low SiO₂ content (basaltic rocks), medium SiO₂ content (schist and granitic rocks), and high SiO₂ content (quartzites), and **b) Carbonate aggregates** (limestone, dolostone and marble). The rocks have been classified according to their composition and origin.

SAMPLES	CATEGORY	SAMPLES	CATEGORY
OPH-1	Silicate aggregate	SCH-1	Silicate aggregate
OPH-2	Silicate aggregate	MYT-1	Silicate aggregate
OPH-3	Silicate aggregate	GRB-1	Silicate aggregate

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OPH-4	Silicate aggregate	GRA-1	Silicate aggregate
OPH-5	Silicate aggregate	GRA-2	Silicate aggregate
AND-1	Silicate aggregate	QRT-1	Silicate aggregate
AND-2	Silicate aggregate	QRT-2	Silicate aggregate
BAS-1	Silicate aggregate	CAL-1	Carbonate aggregate
BAS-2	Silicate aggregate	CAL-2	Carbonate aggregate
MSD-1	Silicate aggregate	CAL-3	Carbonate aggregate
PHI-1	Silicate aggregate	DOL-1	Carbonate aggregate
AMP-1	Silicate aggregate	MAR-1	Carbonate aggregate

Table 1.
Aggregates under
microwave study.



Fig. 1: Samples of 24 aggregates under study.

2.2. Physical characterisation, petrographic analysis, and compositional description

Several tests were carried out to study the physical properties and petrographic, mineralogical, and chemical characteristics. First, the apparent density, described in the standard EN 1936: 2007, was measured [47].

For the petrographic study (textural and compositional description of the rock), the authors followed EN 12407 standard (Natural stone test methods, Petrographic examination) [48]. The terms used in the description are included in the EN 12670 standard (Natural stone. Terminology) [49]. A Zeiss West Germany Optical 316 Microscope with 2.5x, 10x, and 20x magnifications were used for microscopic characterisation with transmitted and polarised light sources. Thin sections, 30 μm -thick, of each rock type were prepared to characterize the mineralogical components.

The point-counting technique has been used to estimate the percentage by volume of minerals forming each sample. It is one of the most common methods for rock classification, for establishing trends and knowing the relative proportions and size distribution of the rock components. Point counting has been made with the software JMicrovision® on pictures of thin sections made with a camera attached to the petrographic microscope. After point counting analysis, measurements of the size (area) and shape of the minerals were made to obtain information on the textural relations and the size of the rock crystals (Fig.2). Finally, a validation of the data was carried out, eliminating the extreme values, and the homogeneity and normality of the data were checked.

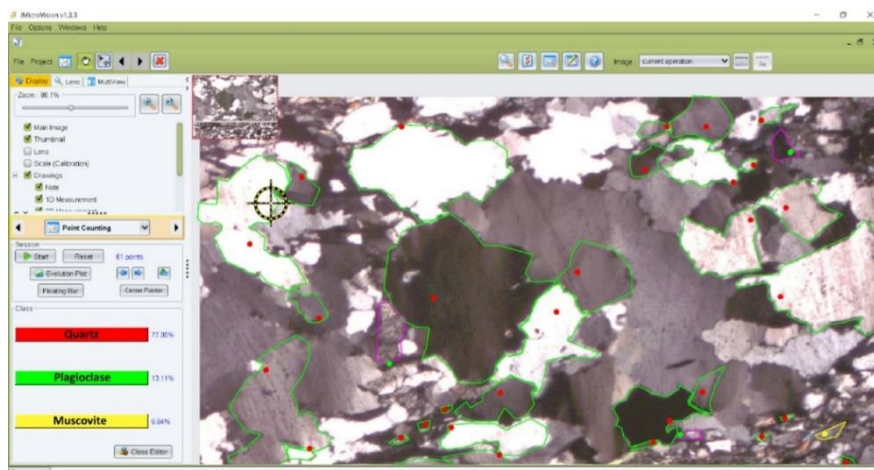


Fig. 2: Sample analysed by JMicrovision®.

1 Bulk mineralogy was determined by X-ray diffraction (XRD). A portion of the
2 samples was ground in an agate mortar to obtain a 53 μm powder. The non-oriented
3 powder was examined with a Philips PW 1720 diffractometer with Cu ($K\alpha$) radiation and
4 a Bruker D8 Advance diffractometer equipped with a Sol-X detector and Cu ($K\alpha$)
5 radiation in the CAI of Geological Techniques (Complutense University of Madrid). The
6 quantification of mineral phases was carried out using the Chung method [50] by Bruker
7 software (EVA[®]). Finally, X-ray fluorescence (XRF) was performed using a wavelength
8 dispersive X-ray fluorescence spectrometer (Bruker, S2 Ranger) to obtain a semi-
9 quantitative analysis of the chemical composition of the sample.
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23 **2.3. Microwave heating**

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27 To study the heating produced by microwave energy in different aggregates, the
28 authors used the T70 protocol proposed in [19], based on the direct measurement of
29 heating in an aggregate sample subjected to microwave radiation. The test measures
30 the time in seconds that a sample of 200 g of aggregate requires to reach 70 °C, from an
31 ambient temperature of 20 °C, by microwave radiation with an output power of 700 W
32 and a frequency of 2.45 GHz.
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40 For this test, the fraction 4/8 mm was used to avoid potential heating differences
41 related to the size of the particles. Before performing the microwave heating, the
42 aggregates were washed and dried in an oven at a temperature slightly below 100 °C.
43 Once the aggregate got dried and reached room temperature again (20 °C), a sample of
44 200g was introduced in a polypropylene plastic container that allows the heating of the
45 aggregate (polypropylene is utterly transparent to microwaves).
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1 An Orbegozo MI-2014 20L microwave oven was used, with an output power of
2 700 W and a frequency of 2.45 GHz (Figure 3); the maximum power was used during
3 the tests. During the T70 test, the temperature of the aggregate surface was measured
4 with an infrared temperature gun (Testo 830-T1), heating the aggregate at fixed time
5 intervals (every 20 seconds) until the bulk aggregate reached a surface temperature
6 above 110 °C.
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31 **Fig. 3:** Equipment used for the heating test.
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36 During the design of the experiment, the randomisation principle was followed
37 with random number tables to ensure the independence of the data. Furthermore, the
38 experiment was repeated 25 times per sample to avoid underestimating the variability.
39 During this process, measurements of the ambient temperature were taken to
40 determine its impact on the results. Subsequently, the data diagnosis was checked to
41 ensure correct (homoscedasticity, normality, and independence). Finally, the average,
42 range and standard deviation were calculated.
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58 **2.4. Permittivity measurements**

59 The dielectric properties of the sample have been determined by the dual-mode
60 microwave system, where the dielectric properties are calculated using a modified
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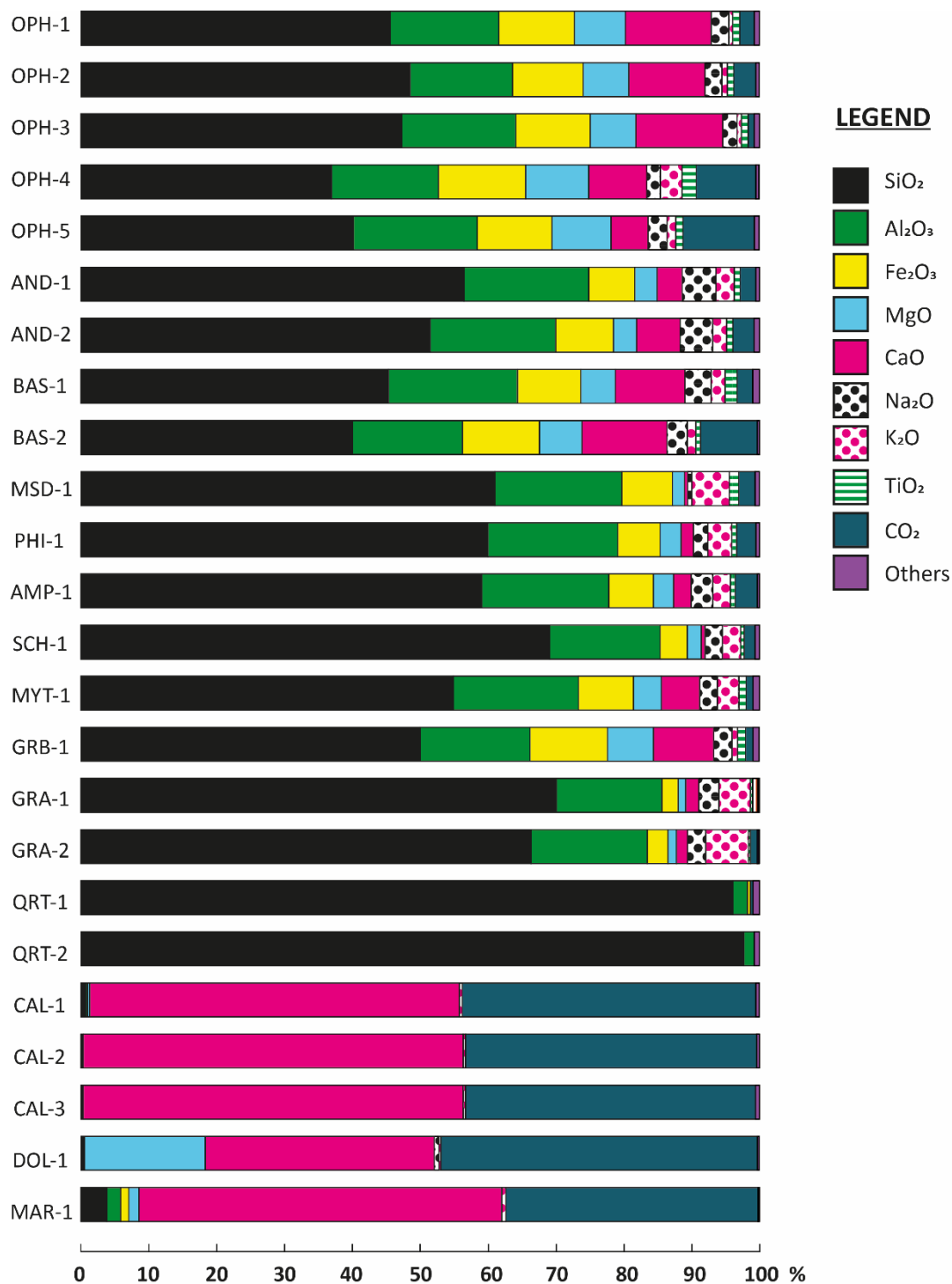


Fig. 4: Chemical data (XRF) for each sample.

Table 3. Chemical data (XRF) for each sample are given in percentage by weight.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	MnO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	SO ₃	Cr ₂ O ₃	CO ₂	Others
OPH-1	45.60	16.03	11.14	7.50	0.06	12.66	2.60	0.54	1.09		0.09	0.08	2.10	0.55
OPH-2	48.50	15.12	10.39	6.77	0.06	11.22	2.52	0.81	0.92		0.09		3.22	0.24
OPH-3	47.30	16.84	10.91	6.74	0.07	12.82	2.08	0.69	0.98		0.09		0.89	0.50
OPH-4	36.90	15.74	12.87	9.30	0.06	8.54	2.01	3.15	2.07	0.27	0.07		8.84	0.16
OPH-5	40.18	18.24	11.03	8.68	0.08	5.44	2.77	1.30	1.06	0.23	0.23		10.56	0.17
AND-1	56.38	18.41	6.78	3.30	0.05	3.70	5.00	2.63	0.86	0.13	0.08		2.30	0.45
AND-2	51.37	18.54	8.51	3.40	0.05	6.42	4.80	2.06	0.93	0.35	0.06		3.10	0.32
BAS-1	45.33	19.00	9.32	5.08	0.06	10.24	3.91	2.06	1.77	0.44	0.07		2.31	0.36
BAS-2	39.92	16.25	11.31	6.23	0.07	12.50	3.02	1.24	0.72	0.05	0.05		8.37	0.17
MSD-1	61.02	18.70	7.51	1.80	0.04	0.46	0.60	5.55	1.36		0.11		2.40	0.40
PHI-1	59.97	19.16	6.22	3.11	0.04	1.81	2.14	3.51	0.75		0.20		2.84	0.21
AMP-1	59.07	18.71	6.53	3.00	0.04	2.60	3.19	2.56	0.75		0.11		3.25	0.19
SCH-1	69.11	16.34	4.00	2.07	0.03	0.57	2.56	2.70	0.51		0.25		1.66	0.19
MYT-1	54.90	18.43	8.13	4.10	0.05	5.67	2.60	3.21	1.15	0.30	0.07		1.00	0.27
GRB-1	49.89	16.18	11.45	6.73	0.07	8.90	2.67	0.85	1.16	0.13	0.58		1.10	0.27
GRA-1	70.09	15.58	2.39	1.09	0.02	1.92	2.98	4.64	0.35		0.05		0.53	0.25
GRA-2	66.37	17.14	3.02	1.29	0.03	1.64	2.67	6.31	0.31				1.03	0.19
QRT-1	96.09	2.15	0.44	0.20		0.07	0.10	0.14	0.10		0.06		0.40	0.16
QRT-2	97.72	1.51	0.19					0.04	0.11		0.03		0.20	0.17
CAL-1	0.72	0.27	0.09	0.30		54.64		0.41			0.05		43.30	0.16
CAL-2	0.35	0.11	0.06	0.23		55.79	0.06	0.34			0.01		42.95	0.09
CAL-3	0.32	0.11	0.09	0.23		56.01	0.01	0.37		0.01	0.01		42.76	0.09
DOL-1	0.37		0.17	17.80	0.01	33.74	0.70	0.27					46.70	0.28
MAR-1	3.78	2.04	1.17	1.51	0.07	53.37	0.06	0.60	0.08		0.03		37.19	0.16

Table 4. Apparent density values in g/cm³.

SAMPLE	PHYSICAL PROPERTIES	SAMPLE	PHYSICAL PROPERTIES
	Apparent density		Apparent density
OPH-1	2.832	SCH-1	2.620
OPH-2	2.784	MYT-1	2.773
OPH-3	2.988	GRB-1	2.758
OPH-4	2.979	GRA-1	2.671
OPH-5	2.877	GRA-2	2.746
AND-1	2.676	QRT-1	2.696
AND-2	2.781	QRT-2	2.713
BAS-1	2.905	CAL-1	2.629
BAS-2	2.873	CAL-2	2.609
MSD-1	2.750	CAL-3	2.650
PHI-1	2.747	DOL-1	2.800
AMP-1	2.743	MAR-1	2.864

Based on the petrographic, mineralogical, and chemical features of the rocks, a classification of the samples has been established. For silicate rocks, two different classifications have been used depending on the genetic origin of the rock. The QAPF diagram of Le Maitre [52] was used to classify igneous rocks according to their modal composition, and the Eskola method modified by Turner and Verhoogen [53] was used for metamorphic rocks. Finally, two classifications have been used for the classification of carbonate rocks: Dunham [54] for rocks of calcitic composition and Friedman [55] for rocks with dolomitic composition.

To facilitate the description of the samples, they were grouped into five categories according to their characteristics, such as mineralogy, chemistry, or texture (Fig. 5). This description will also include the physical characteristics (apparent density and loss tangent).

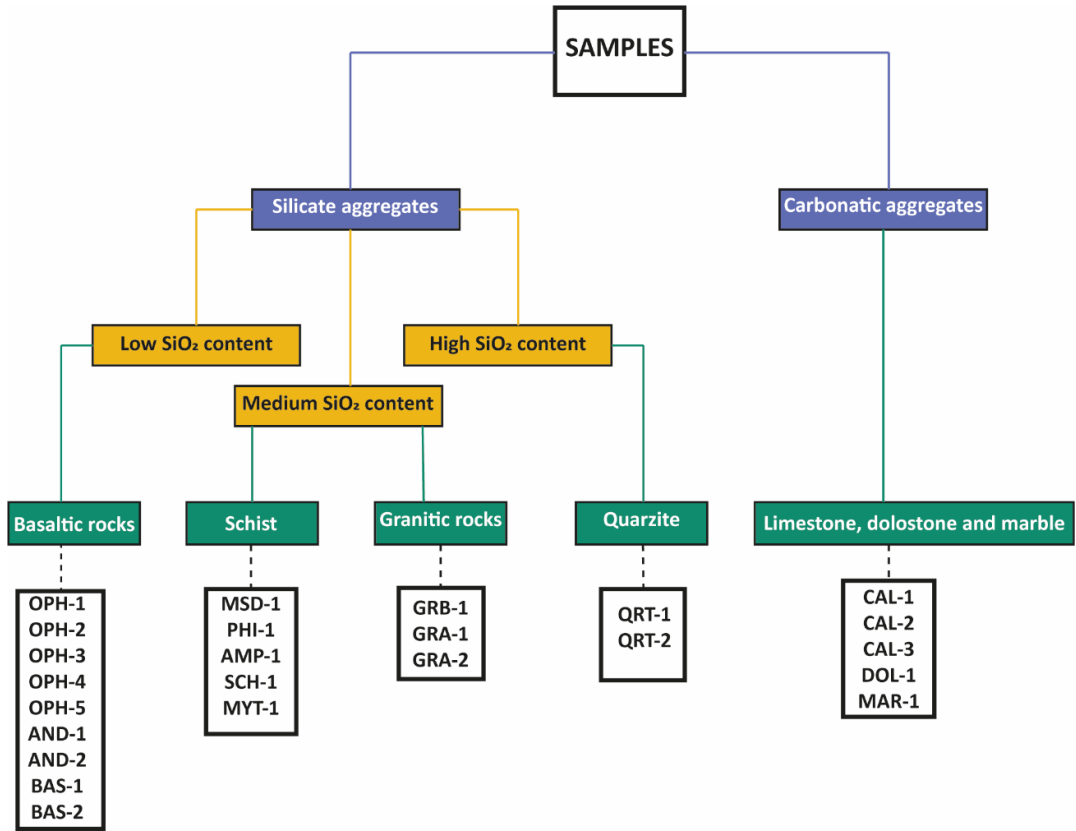


Fig. 5: Samples were studied and their classification according to their composition.

1 A) Silicate aggregates, basaltic rocks (OPH-1, OPH-2, OPH-3, OPH-4, OPH-5,
2 AND-1, AND-2, BAS-1, and BAS-2):
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4 The rocks in this group are volcanic rocks of basic composition with aphanitic or
5 porphyritic textures. The OPH-1, OPH-2, OPH-3, OPH-4, and OPH-5 correspond to
6 diabbases/ophites with plagioclase, augite, and hornblende. In OPH-1 and OPH-3
7 samples, augite minerals form large poikilotopic crystals, encapsulating smaller
8 plagioclase crystals (ophites). The OPH-2, OPH-4, and OPH-5 samples are similar in
9 composition to the previous ones, but the augite crystals do not form poikilotopic
10 crystals (diabbases), both rock groups equigranular texture with a fine-grained
11 intergranular tendency.
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20 XRD results show that the most abundant phases are plagioclase, chlorite,
21 pyroxene (augite), and hornblende. Other minor components are biotite, muscovite,
22 calcite/dolomite, and quartz. The two last minerals correspond to cement from a later
23 stage of rock formation. XRF results show values of SiO₂ between 45-40%, Al₂O₃ 17%,
24 Fe₂O₃ 12%, MgO 7-9%, CaO 12-6%, and TiO₂ 1-2%. The values of CO₂ are low except in
25 the samples OPH-4 and OPH-5 due to the presence of 9% of carbonatic cement.
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33 Finally, the density of the samples ranged between 2.8 -3.0 g/cm³, with higher
34 values in OPH-4 and OPH-5. The loss tangent value at 25 °C for the OPH-1, OPH-2, OPH-
35 3, and OPH-5 samples are 0.015, and for the OPH-4 sample, it is near 0.03.
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40 The samples AND-1 and AND-2 correspond to andesite, with a fine-medium grain
41 hypocrySTALLINE texture. The predominant mineral phases correspond to plagioclase,
42 hornblende, and volcanic glass. Finally, the BAS-1 and BAS-2 samples correspond to
43 basalts with aphanitic (microcrystalline) texture. The predominant phases are anorthite,
44 amphibole, chlorite, and olivine.
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50 XRD results for andesite and basalt samples are very similar. The most abundant
51 phases are plagioclase with values between 30-50%, hornblende >30%, chlorite, olivine
52 (only in basalt), biotite, quartz (volcanic glass), and calcite cement. The XRF results show
53 variable percentages of the different components with 40-54% SiO₂ values, higher in the
54 andesites, Al₂O₃ 16-19%, Fe₂O₃ 7-11%, MgO 3-6%, CaO 4-13%, Na₂O 3-5%, K₂O 2%, and
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1 TiO₂ 1%. The BAS-1 and BAS-2 samples present a higher CaO value due to the carbonate
2 cement formation formed during a hydrothermal process.
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4 Finally, the density is 2.6-2.7 g/cm³ for andesite and 2.8-2.9 g/cm³ for the basalt
5 samples. The loss tangent at 25 °C ranges from 0.028-0.022 for the andesite samples
6 and 0.017 for the basalt samples.
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16 *B) Silicate aggregates, schist rocks (MSD-1, PHI-1, AMP-1, SCH-1, and MYT-1):*
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18 The rocks of this group are metamorphic with a foliated texture and a silica
19 content ranging from 50% to 70%. Sample SCH-1 corresponds to schist, and MSD-1
20 corresponds to a combination of schist and andesite. Both have a lepidoblastic texture
21 of medium grain, and the predominant minerals are quartz and minerals from the mica
22 group. The PHI-1 sample corresponds to phyllite with a fine-grained lepidoblastic
23 texture, and the predominant minerals are quartz and minerals from the mica group
24 (biotite and chlorite). The sample MYT-1 corresponds to mylonite, a rock formed by
25 dynamic metamorphism, with a fine-grained granular texture, a high content of quartz,
26 and a lower content of other minerals such as plagioclase and muscovite. Finally, the
27 AMP-1 sample corresponds to a fine-grained amphibolite with decused texture and
28 abundant chlorite due to hydrothermal activity.
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41 The results obtained for the mineralogy (XRD) of all samples of this group show
42 that they have the typical mineralogy for these lithologies, with variable values of quartz,
43 plagioclase, muscovite, and chlorite. The XRF results show that the most abundant
44 element is SiO₂ with values between 55-70%; the rest of the elements are similar
45 between the different aggregates with approximate values of Al₂O₃ 17%, Fe₂O₃ 7%, MgO
46 3%, K₂O 3%, and Na₂O, and TiO₂ values between 0.5-1.5%.
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53 The density of these samples is 2.7 g/cm³, and the loss tangent value at 25 °C is
54 less than 0.01 for SCH-1, PHI-1, AMP-1, AMP-1, and while for MSD-1, the value is 0.0141.
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C) Silicate aggregates, granitic rocks (GBR-1, GRA-1, and GRA-2):

The rocks of this group are plutonic rocks of acidic or intermediate composition. The samples GRA-1 and GRA-2 correspond to granites with a similar composition, predominantly quartz, feldspar, and biotite with a granite texture of the medium-fine grain. The GRB-1 sample corresponds to diorite. The predominant minerals in GRB-1 are quartz, hornblende, and plagioclase, with a granite texture of the medium-fine grain.

XRD results show that the GRB-1 rock has approximately 30% quartz, while 55% of GRA-1 and GRA-2 samples are quartz. Other differences between the samples are 10-20% K-feldspar in the GRA-1 and GRA-2 samples and the presence of 30% hornblende in GRB-1. The rest of the phases are similar in all samples: plagioclase, biotite, and chlorite. The FRX results show significant differences in some elements between the rocks GRB-1, GRA-1, and GRA-2, the content of silica is 70% in the case of granites and 50% for diorite. In the GRB-1, percentages of Fe₂O₃ are 12%, Al₂O₃ 16%, MgO 7%, CaO 9%, and TiO₂ 1%. In the case of GRA-1 and GRA-2 are Al₂O₃ 16%, Fe₂O₃ 3%, MgO 1%, CaO 2%, K₂O 5-6%, and TiO₂ <0.5%.

Finally, the apparent density of the samples is 2.7 g/cm³, and the value of the loss tangent is less than 0.01 for GRB-1 and GRA-1, but the sample GRB-2 value is 0.0152 at 25 °C.

D) Silicate aggregates, quartzites (QRT-1 and QRT-2):

Non-foliated metamorphic rocks (granular texture) with a high silica content form this group, corresponding to quartzites with a quartz content above 95%. The difference between both samples is the crystal size: QRT-1 has fine-medium size crystals, while the crystals in QRT-2 are coarser.

XRD results show that the only mineral phase present is quartz. The XRF results for both samples are similar, with the content of SiO₂ greater than 96% and about 2% of Al₂O₃.

The density of the samples is 2.7 g/cm³, and the value of the loss tangent is less than 0.005 at 25 °C.

E) *Carbonatic aggregates (CAL-1, CAL-2, CAL-3, DOL-1, and MAR-1):*

This group consists of sedimentary rocks of carbonatic composition, except the sample MAR-1 that is metamorphic. CAL-1 and CAL-2, 3 correspond to limestones with micritic texture and sparitic cement in porosity, CAL-3 correspond to limestones with grainstone texture and sparitic cement in porosity. Sample DOL-1 corresponds to a dolostone, and sample MAR-1 to a calcite and dolomite marble with a fine-grained crystalline texture.

XRD results show that the predominant mineral phases are calcite, dolomite, and traces of quartz. The FRX results show that the CaO values are around 55%, except in the case of dolomite, with 34% CaO and 17% MgO. In addition, the MAR-1 aggregate presents other components in its composition, such as SiO₂ 4%, Al₂O₃ 2%, Fe₂O₃ 1%, and MgO 1%.

Finally, the density of the limestone samples is 2.6 g/cm³, and for the samples of dolomite and marble, 2.8 g/cm³. Concerning the loss tangent, the values are below 0.01 at 25 °C.

3.2. Crystal size assessment

The measurement area of the minerals present in each aggregate has been collected. The values obtained for the crystal sizes are between 0.001 and 1.5 mm²; this allows us to observe the differences between the crystal sizes in each group of rocks. The minerals with a total content below 5% of the rock volume have been omitted.

Regarding the average crystal size, rocks can be classified into three groups: coarse, medium, and fine. The coarse-grained group comprises granites, quartzite (QRT-2), and marble, with mean values above 0.2 mm². The group of medium-grained rocks is formed by samples of andesite, mylonite, granodiorite, and quartzite (QRT-1), all with average values between 0.025 and 0.2 mm². Finally, the fine-grained rocks group consists of samples of ophite, basalt, meta-sandstone, phyllite, amphibolite, schist, carbonate, and dolomite, with values below 0.025 mm².

A detailed study has been carried out, differentiating the minerals of each rock and indicating in which rocks each mineral is found. To get this diagram, the measurement area of each mineral in different aggregates has been collected (Fig.6). The values obtained are between 0.001 and 1.5 mm² for the different samples studied to observe differences between minerals. A total of 11 minerals have been differentiated: biotite, hornblende, pyroxene, calcite/dolomite, K-feldspar, chlorite, olivine, muscovite, quartz, plagioclase, and iron oxides. The minerals have been compared to each other. A description of the results obtained for the minerals studied by crystal size has been made in Table 5.

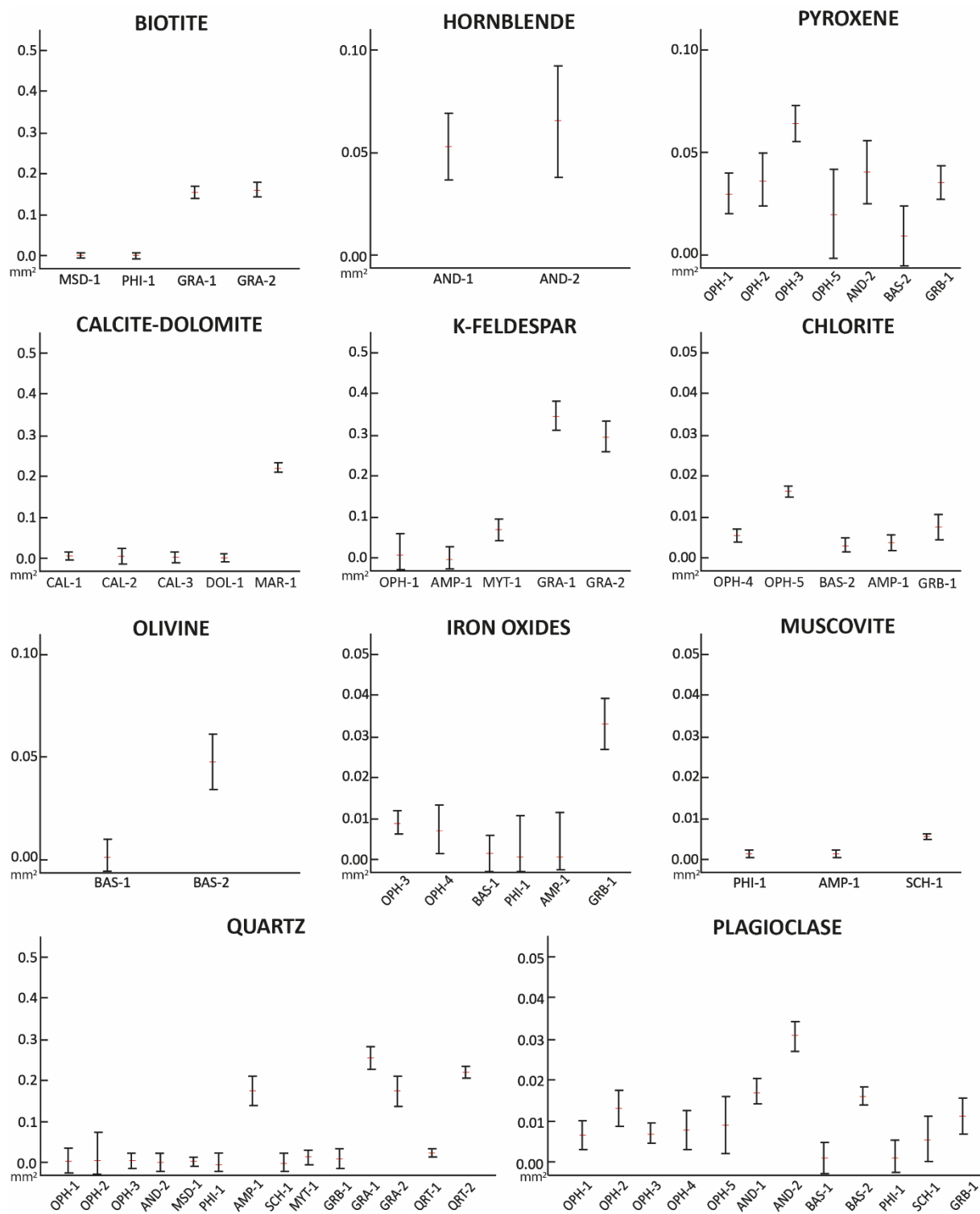


Fig. 6: Distribution of crystal size in different minerals.

Table 5. Summary of crystal size of minerals.

Mineral	Rock	Crystal size
Biotite	- Foliated (MSD-1 and PHI-1) - Granitic (GRA-1 and GRA-2)	Coarse grain: GRA-1 and GRA-2 Fine-grain: MSD-1 and PHI-1
Hornblende	Basaltic (AND-1 and AND-2)	Fine-grain: AND-1 and AND-2
Pyroxene	- Basaltic (OPH-1, OPH-2, OPH-3, OPH-5, AND-2, and BAS-2) - Granitic (GRB-1)	Medium-fine grain: OPH-1, OPH-2, OPH-3, OPH-5, AND-2, and GRB-1 Fine-grain: BAS-2
Calcite/Dolomite	Carbonatic (CAL-1, CAL-2, CAL-3, DOL-1, and MAR-1)	Coarse grain: MAR-1 Fine grain: CAL-1, CAL-2, CAL-3, and DOL-1
K-Feldspar	- Basaltic (OPH-1) - Foliated (AMP-1 and MYT-1) - Granitic (GRA-1 and GRA-2)	Coarse grain: GRA-1 and GRA-2 Fine-grain: OPH-1, AMP-1, and MYT-1
Chlorite	- Basaltic (OPH-4, OPH-5, and BAS-2) - Foliated (AMP-1) - Granitic (GRB-1)	Coarse grain: OPH-5 Medium-fine grain: OPH-4, BAS-2, AMP-1, and GRB-1
Olivine	Basaltic (BAS-1 and BAS-2)	Coarse grain BAS-1 Fine-grain: BAS-2
Muscovite	Foliated (PHI-1, AMP-1, and SCH-1)	Fine-grain: PHI-1, AMP-1, and SCH-1
Quartz	- Basaltic (OPH-1, OPH-2, OPH-3, and AND-2) - Foliated (MSD-1, PHI-1, AMP-1, SCH-1, and MYT-1) - Granitic (GRB-1, GRA-1, and GRA-2) - Quartzites (QRT-1 and QRT-2).	Coarse grain: QRT-2, GRA-1, and GRA-2 Fine grain: OPH-1, OPH-2, OPH-3, AND-2, MSD-1, PHI-1, AMP-1, SCH-1, MYT-1, GRB-1, and QRT-1
Plagioclase	- Basaltic (OPH-1, OPH-2, OPH-3, OPH-4, OPH-5, AND-1, AND-2, BAS-1, and BAS-2) - Foliated (PHI-1 and SCH-1) - Granitic (GRB-1).	Coarse grain: AND-2 Medium-fine grain: OPH-2, AND-1, and BAS-2 Fine-grain: OPH-1, OPH-3, OPH-4, OPH-5, BAS-1, PHI-1, SCH-1 and GRB-1
Iron oxides	- Basaltic (OPH-3, OPH-4, AND-2, and BAS-1) - Foliated (PHI-1 and AMP-1) - Granitic (GRB-1).	Medium grain: GRB-1 Fine-grain OPH-3, OPH-4, AND-2, BAS-1, PHI-1, and AMP-1

3.3. Analysis of microwave heating

The microwave heating results obtained have been used to calculate the value of T70 for each aggregate, classifying them into five classes. The first class corresponds to samples with faster heating: from 20 °C to 70 °C in less than 25 seconds. The second class includes samples that require between 25 and 50 seconds. The third class with samples that presented medium heating, passing from 20 °C to 70 °C between 50 and 100 seconds. The fourth class samples (slower heating) need between 100 and 200 seconds. Finally, the fifth class includes the samples that require more than 200 seconds to pass from 20 °C to 70 °C.

Table 6 shows the T70 values for each sample. The samples with high microwave susceptibility (HS), with T70 between 25 and 50s, are both andesites (AND-1 and AND-2). Samples between 50 and 100 seconds (medium susceptibility, MS) belong to multiple groups: subvolcanic rocks (OPH-1, OPH-2, OPH-3, OPH-4, and OPH-5), volcanic rocks, corresponding to basalts (BAS-1 and BAS-2); and the last group, foliated metamorphic rock with a sample (MSD -1). The T70 class samples between 100 and 200 seconds (low susceptibility, LS) belong to multiple groups: plutonic rocks (GRB-1, GRA-1, and GRA-2), metamorphic rocks (foliated) with different degrees (PHI- 1, AMP-1, and MYT-1) and sedimentary rock (DOL-1). Finally, the group of rocks that have a very low susceptibility (VLS) to microwaves (T70> 200s) are foliated metamorphic rocks (SCH-1), non-foliated metamorphic rocks: river-gravel quartzite (QRT-1), crushed quartzite (QRT-2), and marble (MAR-1).

Table 6. T70 values for samples.

SAMPLES	T70 (s)	SAMPLES	T70 (s)
OPH-1	57.7	SCH-1	245.4
OPH-2	97.2	MYT-1	153.4
OPH-3	75.1	GRB-1	113.9
OPH-4	65.8	GRA-1	161.8
OPH-5	78.2	GRA-2	118.8
AND-1	42.3	QRT-1	289.7
AND-2	47.4	QRT-2	504.2
BAS-1	72.2	CAL-1	334.5
BAS- 2	67.7	CAL-2	310.9
MSD-1	86.7	CAL-3	426.7
PHI-1	112.7	DOL-1	123.5
AMP-1	120.6	MAR-1	351.4

The temperature rise of the sample during microwave irradiation is linear with energy consumption [14], so the slope of the line represents the susceptibility of the material to microwaves. The steeper the slope of the line, the greater the susceptibility to microwaves and the more efficient the process. The calculated slopes are presented in Figure 7. It can be seen that the rocks of andesite, ophite, and basalt are the samples most susceptible to microwaves.

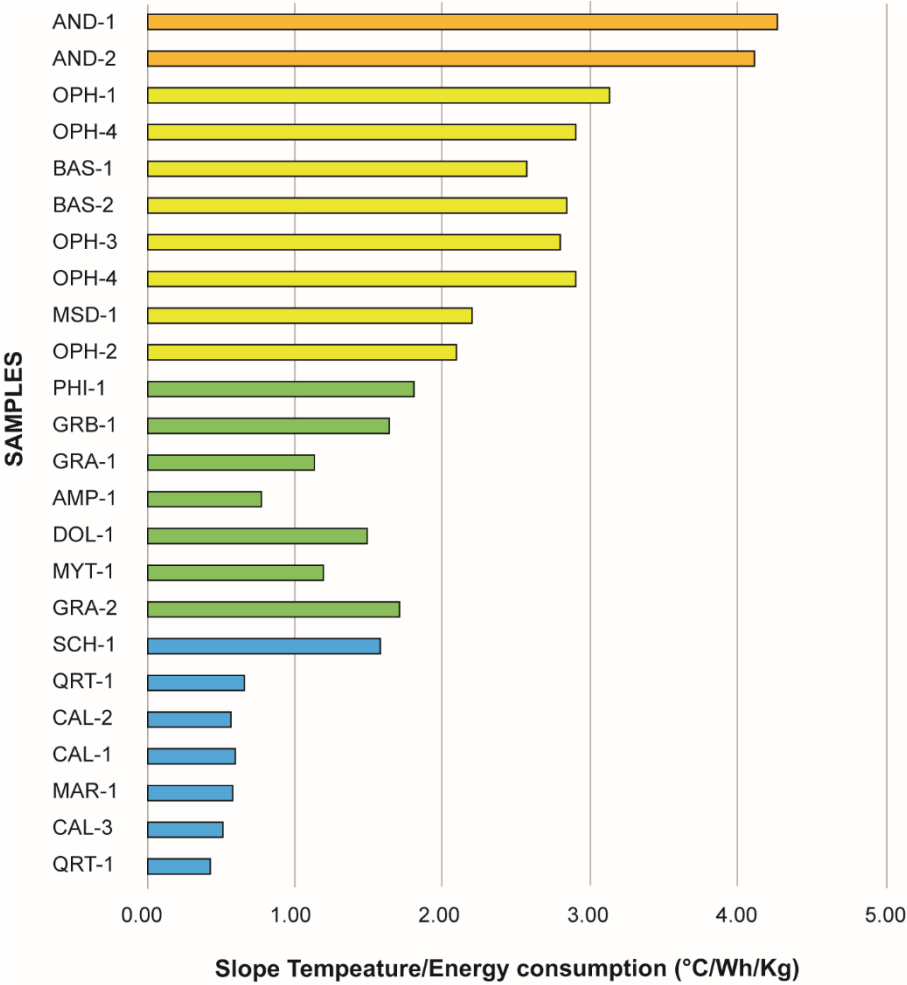


Fig.7: Slope of temperature versus energy consumption (susceptibility to microwaves). Samples were ordered from lowest to highest T70.

3.4. Results of the dielectric parameters

It has been reported that the dielectric permittivity values have a high relationship with the microwave susceptibility **values of** aggregates [19]. Therefore, in Table 7, the values of permittivity obtained in this study at 25 °C employing the CPM technique are shown. The values of the dielectric constant (ϵ'), loss factor (ϵ''), and loss tangent are included. The loss factor is calculated as the relationship between the loss factor and the dielectric constant (ϵ''/ϵ').

Table 7. Permittivity values.

SAMPLE	DIELECTRIC PROPERTIES		
	ϵ'	ϵ''	ϵ''/ϵ'
OPH-1	2.5949	0.0353	0.0136
OPH-2	3.0512	0.0463	0.0152
OPH-3	3.3398	0.0479	0.0143
OPH-4	2.7613	0.0804	0.0291
OPH-5	2.4578	0.0262	0.0107
AND-1	2.4380	0.0686	0.0281
AND-2	2.5275	0.0574	0.0227
BAS-1	3.3053	0.0562	0.0170
BAS-2	3.0203	0.0516	0.0171
MSD-1	2.6045	0.0366	0.0141
PHI-1	2.5752	0.0175	0.0068
AMP-1	2.7260	0.0144	0.0053
SCH-1	2.5756	0.0195	0.0076
MYT-1	2.5855	0.0160	0.0062
GRB-1	3.0580	0.0255	0.0083
GRA-1	2.1630	0.0132	0.0061
GRA-2	2.0679	0.0313	0.0152
QRT-1	1.9863	0.0061	0.0031
QRT-2	2.3947	0.0043	0.0018
CAL-1	3.3373	0.0132	0.0040
CAL-2	3.0184	0.0163	0.0054
CAL-3	3.2083	0.0141	0.0044
DOL-1	3.1244	0.0103	0.0033
MAR-1	3.0274	0.0237	0.0078

4. Discussion

Different studies have shown that the differential heating of materials with microwaves could be caused by various physical characteristics (crystal size and density) or compositional characteristics (mineralogy and chemistry). In the studies of [14,19], aggregates with different compositions were tested, and it was observed, using the parameter T70, the existence of differential heating among aggregates of different nature. The study [19] revealed that measurements of dielectric permittivity (loss tangent) provide results about the susceptibility to the microwave of aggregates similar to those obtained with the parameter T70, displaying a high correlation ($R^2 = 0.83$). To determine the factors influencing the heating response, the heating data (parameter T70) and dielectric permittivity (loss tangent) values should be compared with the values of mineralogy, chemistry, crystal size, and apparent density to observe the influence of these factors.

The minerals and elements detected in the XRD and XRF tests were compared with the dielectric permittivity (loss tangent) and microwave heating data (parameter T70). The resulting data were used for several regression models as described in the following sections.

4.1. Relationship of mineralogical, chemical, and physical properties with loss tangent and microwave heating (T70)

Mineralogical composition

From the results obtained by XRD, 17 different mineral phases (formation and alteration minerals) were obtained, only 11 mineral phases have been studied because only the formation minerals have been considered, eliminating alteration minerals and minerals with a low proportion (<5%). Silicate minerals including quartz, plagioclase, feldspar, biotite, muscovite, chlorite, hornblende, pyroxene, and olivine have been studied. The carbonate minerals included in the study were calcite and dolomite. Finally, olivine and dolomite minerals have been removed due to insufficient data.

Correlations between minerals and loss tangent are low (Table 8) due to the absence of correlation or the low presence of some minerals (olivine and dolomite). However, two strong correlations are observed for muscovite and calcite, with negative slope equations and high R^2 , indicating that a higher presence of these minerals reduces the loss tangent values as previously reported [23].

In the same line, the 11 most relevant mineral phases have been correlated with the parameter T70. Strong heating correlations have been found in the minerals studied with pyroxene and muscovite and moderate correlations in the case of quartz (Table 8). Summarising, based on the sign of the slope of the equation, muscovite and quartz reduced the heating, while pyroxene promoted the heating potential of microwaves. Quartz, muscovite, and pyroxene behaviour is consistent with the findings of other authors [12,21,26,56].

In other minerals, moderate to weak correlations have been obtained, and this may be due to different causes, such as concentrations of similar minerals between different rocks or low mineral content in the rock. Therefore, finding moderate correlations due to these causes does not indicate that the mineral influences microwave heating.

Table 8. Equations and Pearson's correlation coefficients for minerals versus loss tangent and T70 parameter.

Minerals	ϵ''/ϵ'		T70	
	Equation	R^2	Equation	R^2
Quartz	$y = -8E-05x + 0.0144$	0.16	$y = 2.7322x + 30.551$	0.53
Biotite	$y = -0.0004x + 0.0146$	0.03	$y = 7.8573x + 71.769$	0.27
Hornblende	$y = 0.0001x + 0.0153$	0.03	$y = -0.2543x + 78.986$	0.01
Plagioclase	$y = 0.0001x + 0.01$	0.07	$y = -0.2565x + 110.22$	0.01
Muscovite	$y = -0.0001x + 0.0142$	0.91	$y = 0.747x + 74.395$	0.79
K-feldspar	$y = 0.0006x + 0.0058$	0.24	$y = -2.2314x + 156.3$	0.20
Chlorite	$y = 0.0002x + 0.01$	0.17	$y = -2.3553x + 137.38$	0.29
Pyroxene	$y = -8E-05x + 0.0146$	0.09	$y = -1.8129x + 103.56$	0.71
Calcite	$y = -0.0002x + 0.0277$	0.83	$y = 0.6627x + 292.11$	0.01

Chemical composition

The results obtained from dielectric permittivity and chemical composition by XRF have been compared with linear regression models and multiple correlation diagrams. Only nine elements were studied from the XRF results, eliminating elements with a low proportion (trace chemical compounds). The chemical compounds studied are SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O , TiO_2 , and MnO . During the analysis, silicate rocks and carbonate rocks were studied separately to know the impact of CaO or MgO on silicate rocks and carbonate rocks, respectively (Table 9). Moderate correlations with $\varepsilon''/\varepsilon'$ have been found with SiO_2 , Fe_2O_3 , and TiO_2 . In the case of SiO_2 , a greater presence of this compound results in a reduction in the loss tangent values, by contrast, in the Fe_2O_3 and TiO_2 compounds, an increase in the loss tangent values is observed. In this case, the results found are consistent with previous studies [23].

A similar study was carried out, correlating the mentioned chemical compounds obtained by XRF and the T70 parameter (Table 9). For silicate rocks, high-moderate correlations of elements that promote heating (with a negative slope of the T70 correlation) have been found for Al_2O_3 , Fe_2O_3 , as well as for CaO , MgO , TiO_2 , and MnO . On the other hand, SiO_2 negatively affects heating in silicate rocks, as the slope of the correlation line is positive. For carbonatic rocks, the compounds CaO and MgO have been studied, both showing a high correlation, and it is observed that CaO affects negatively heating (positive slope of the T70 correlation), and MgO affects it positively (negative slope). The results obtained on the CaO are interesting since all studies carried out so far indicated that this element negatively affects heating [28,30,31]. This result is because they only studied carbonate rocks or carbonate minerals. Nevertheless, it should be noted that calcium was never taken into account for silicates, and this element is present in several minerals, such as pyroxene.

Table 9. Equations and Pearson's correlation coefficients for the chemical compounds described by XRF for loss tangent and parameter T70.

Chemical compounds	ϵ''/ϵ'		T70	
	Equation	R ²	Equation	R ²
Fe ₂ O ₃	$y = 0.0011x + 0.0044$	0.31	$y = -21.271x + 291.27$	0.57
SiO ₂	$y = -0.0003x + 0.0295$	0.39	$y = 5.6815x - 195.5$	0.74
Al ₂ O ₃	$y = 0.0007x + 0.002$	0.20	$y = -18.436x + 421.37$	0.71
Na ₂ O	$y = 0.003x + 0.0052$	0.23	$y = -29.676x + 190.98$	0.28
K ₂ O	$y = 0.0007x + 0.0112$	0.02	$y = -15.699x + 168.5$	0.06
TiO ₂	$y = 0.0091x + 0.0046$	0.35	$y = -135.3x + 252.85$	0.39
MnO	$y = 0.1275x + 0.0074$	0.08	$y = -1789.7x + 193.64$	0.35
CaO (silicates)	$y = -0.0001x + 0.0138$	0.14	$y = -8.4195x + 161.61$	0.34
MgO (silicates)	$y = 0.0011x + 0.0084$	0.16	$y = -14.197x + 174.88$	0.35
CaO (carbonates)	$y = 9E-05x + 0.0006$	0.22	$y = 10.962x - 246.46$	0.86
MgO (carbonates)	$y = -0.0001x + 0.0054$	0.23	$y = -13.448x + 363.38$	0.85

Physical properties: the crystal size

A factor of differential heating found in the literature is the size of the crystals that form the rock, giving rise to heterogeneity in the sample and causing deficiencies in heat transmission [26,27]. Therefore, the results obtained from the dielectric permittivity (loss tangent) and the crystal size data obtained by the imaging technique have been compared to analyse the influence of crystal size.

The correlations observed in this study are low, obtaining inconclusive results due to the interference of multiple minerals. It has only been possible to establish correlations in mono-mineral rocks. As there is no interference between the different mineral phases, moderate correlations have been obtained, which indicate a reduction in the tangent values of loss / increase of T70 in calcite and quartz minerals with a larger crystal size (Fig. 8). Nicco [46] studies carried out on granites provided similar results to those found in this work.

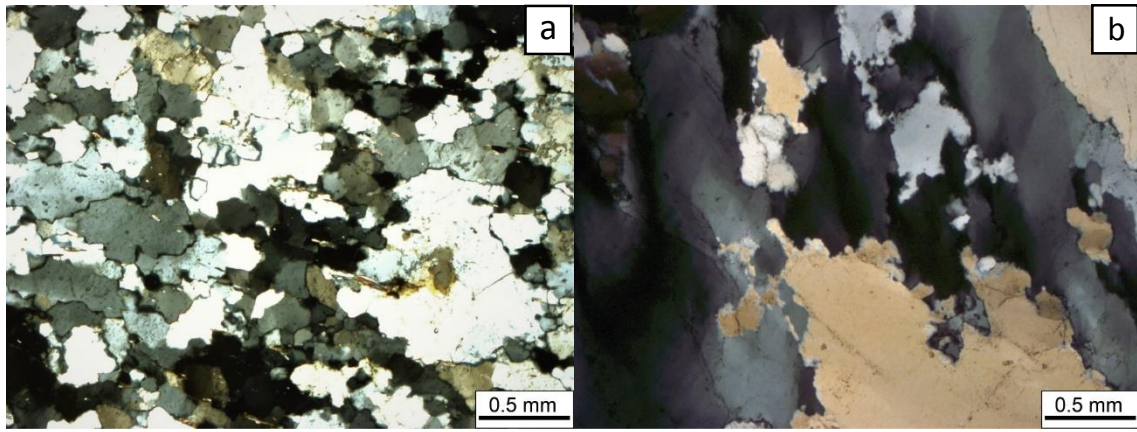


Fig. 8: Images of thin sections (crossed nicols) of **a)** quartzite-1 (QRT-1) and **b)** quartzite-2 (QRT-2).

The samples QRT-1 and QRT-2 are made up of quartz in more than 95%, which is why they are considered quartz mono-mineral rocks. The only discrepancy between them is the crystal size with a mean size for the QRT-1 sample of 0.1 mm^2 and the QRT-2 sample of 0.25 mm^2 . After this, there are discrepancies in the T70 parameter, obtaining values of 289.7 for QRT-1 and 504.2 for QRT-2. It is observed that **rocks with a larger crystal size experience longer heating times for the same composition**. Something similar can be observed in the samples CAL-1, CAL-2, and CAL-3. All of them are micritic limestones (microcrystals) with different proportions of sparite crystals with a size of 0.05 mm^2 . The CAL-1 and CAL-2 samples have 15-20% sparite and the CAL-3 sample 30%. As in quartzites, discrepancies have been observed in the T70 values for the samples with values of 310.9 for CAL-1, 334.5 for CAL-2, and 426.7 for CAL-3.

Physical properties: apparent density

The data of the apparent density for the 24 samples studied were related to the dielectric permittivity (loss tangent). In this case, a linear regression model has been performed (Fig. 9). The correlation between density and dielectric permittivity (loss tangent) is low, and there is no correlation between these two parameters.

Moreover, the relationship between density and heating of materials with microwaves has been studied. As in the previous case, the results obtained from heating with microwaves (T70) and the apparent density data have been compared, performing a linear regression model (Fig. 10). The correlation between density and heating is low,

and there is no relationship between these parameters, contrary to what has been reported by other authors [38]. This discrepancy may be due to several reasons, including the test method or the samples used. In this study, mainly silicate samples in powder format have been used, while in other studies [38], a mixture of silicate and metallic minerals were tested.

Some studies [33] indicate that density is one parameter that affects the loss tangent; these results can be altered in materials with low porosity. Porosity can promote heating since the pores can contain water in different states [21,40]. In the case of road aggregates, porosity is harmful and normally avoided in road pavements.

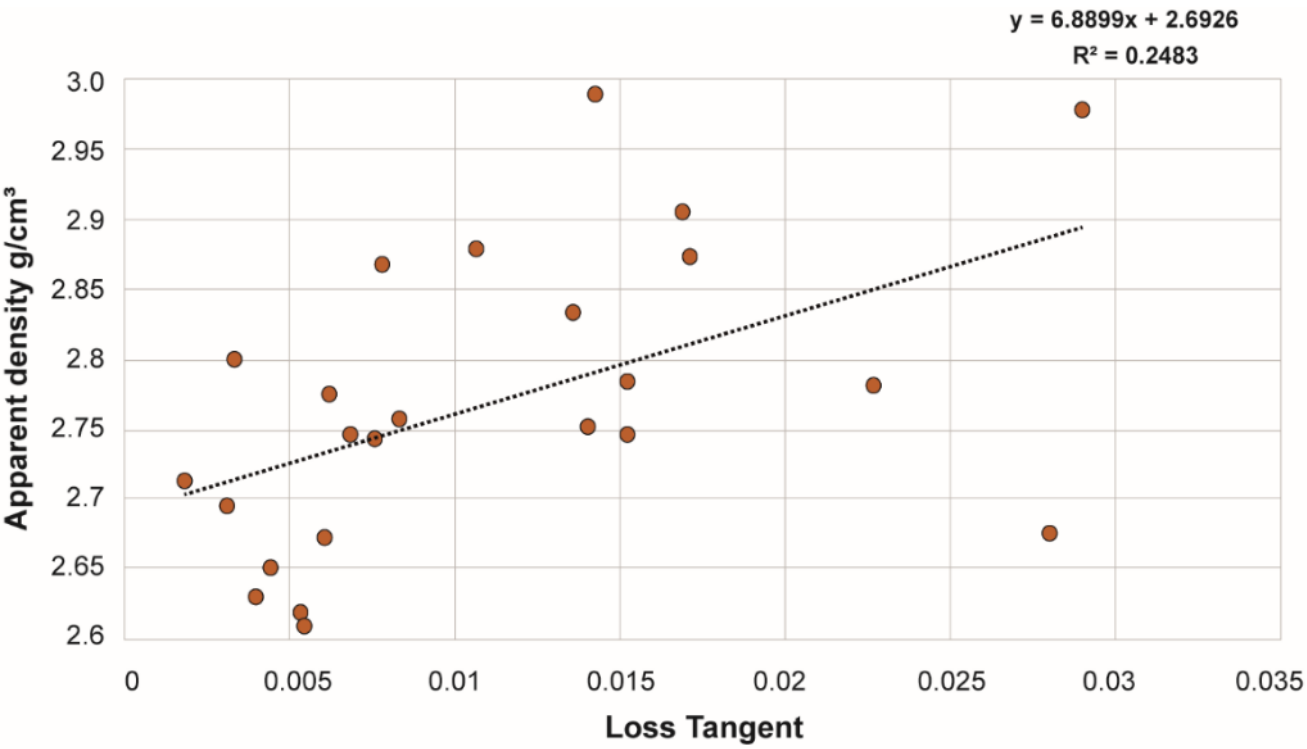


Fig. 9: Dielectric permittivity values versus apparent density.

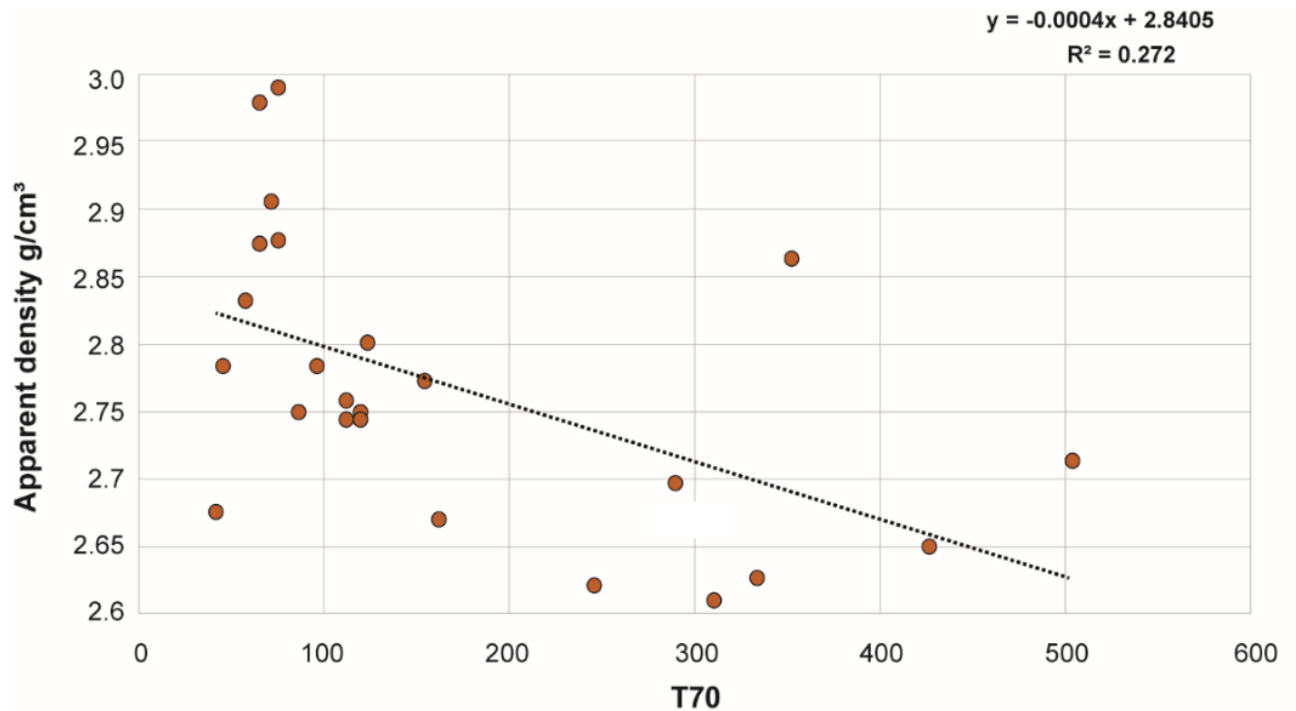


Fig. 10: T70 values versus apparent density.

4.2. A model to predict the microwave susceptibility of the rocks based on their composition

Multiple regression analyses have been performed to apply this knowledge to any rock susceptible to being used as aggregate for asphalt mixtures. The search for the models has been carried out for two microwave susceptibility parameters: parameter T70 and the loss tangent. The chemical and physical properties were analysed as possible causes of differential heating. The most likely causes of differential heating are chemistry and mineralogy. For this reason, models will be made for chemistry and mineralogy, leaving aside the crystal size and the apparent density.

The first two models were made for direct heating data (parameter T70): the first for mineralogical data obtained with the XRD test and the second for chemical elements obtained with the XRF test. With these models, it would be possible to predict the microwave susceptibility that any rock has, including it in one of the categories of parameter T70.

For the study of the influence of mineralogy on heating, eleven variables (minerals) have been used in the model, which are quartz, plagioclase, k-feldspar, biotite, muscovite, chlorite, hornblende, pyroxene, olivine, calcite, and dolomite.

Some of the variables were discarded from the study because they presented autocorrelation with other variables; they were a linear combination of them or were not statistically significant. As a result, only the following minerals were introduced in the model: calcite (Cal), quartz (Qz), chlorite (Chl), pyroxene (Px), and hornblende (Hbl). As a result, the adjusted coefficient of determination (R^2) obtained is 0.7963, and the equation obtained is the following:

$$T70 = 114.376 + 2.58537 \times Cal + 0.575402 \times Qz - 1.22743 \times Chl - 0.952189 \times Px - 1.74457 \times Hbl \quad (2)$$

A similar process has been followed with XRF data to obtain a model for the chemical data; 10 variables (elements) analysed during the phase of the influence of each element have been used in the model, which are SiO_2 , Al_2O_3 , Fe_2O_3 , CaO, MgO, Na_2O , K_2O , TiO_2 , and MnO.

Some of the variables were removed from the model due to a high autocorrelation with other variables due to a linear combination of these or because they had no statistically significant relevance. Nevertheless, the following elements are part of the model: carbonate (CaO), silica (SiO_2), aluminium (Al_2O_3), and iron (Fe_2O_3). The adjusted coefficient of determination (R^2) obtained is 0.8633, and the equation obtained is the following:

$$T70 = 300.827 + 1.11453 \times CaO + 0.22899 \times SiO_2 - 7.52744 \times Al_2O_3 - 10.6784 \times Fe_2O_3 \quad (3)$$

Next, the same process was carried out for the loss tangent. The analyses were performed for the mineralogical data obtained with an XRD test and chemical elements obtained with an XRF test. For the study of mineralogy, the 11 same variables have been used to calculate the model for the parameter T70. Part of the variables was discarded

from the model since they were not statistically significant or presented autocorrelation with other variables. Therefore, they have been used: plagioclase (Plg), muscovite (Ms), chlorite (Chl), and pyroxene (Px). The adjusted coefficient of determination (R^2) obtained is 0.5135, and the equation obtained is the following:

$$\begin{aligned} \text{Loss Tangent} = & 0.00484136 + 0.000191515 \times \text{Plg} - 0.000164382 \times \text{Ms} + \\ & 0.000443923 \times \text{Chl} - 0.00023827 \times \text{Px} \end{aligned} \quad (4)$$

The same process has been followed to study the variables chemical elements. Some variables were eliminated from the model due to the lack of statistical significance or high autocorrelation with other variables. Nevertheless, the following elements are part of the model: silica (SiO_2), aluminium (Al_2O_3), iron (Fe_2O_3), sodium (Na_2O), and potassium (K_2O). The adjusted coefficient of determination (R^2) obtained is 0.6544, and the equation obtained is the following:

$$\begin{aligned} \text{Loss Tangent} = & 0.00415403 - 0.00000152257 \times \text{SiO}_2 - 0.00214932 \times \text{Al}_2\text{O}_3 \\ & + 0.00247492 \times \text{Fe}_2\text{O}_3 + 0.00553521 \times \text{Na}_2\text{O} + 0.00425194 \times \text{K}_2\text{O} \end{aligned} \quad (5)$$

The best correlations between the different physical and chemical properties have been obtained with the direct heating method (parameter T70). The models elaborated with the permittivity values could also be used since moderate correlation coefficients are obtained. In addition, other studies have shown a high correlation between the T70 parameter and the loss tangent values [19].

4.3. Estimation of the influence of physical and chemical properties of the rocks on microwave heating

The models made with the T70 parameter and the loss tangent allow us to observe that higher correlations are obtained with the T70 parameter (Table 10). This result is advantageous since this measurement technique is easy to apply and is a rapid and low-cost technology. With the models carried out for chemistry and mineralogy with the T70 parameter, 86.33% and 79.63% of the variability present in the T70 can be explained. However, other variables such as crystal size, thermal conductivity [57,58],

and electrical conductivity [59,60] of the aggregates can affect heating. From the coefficients of determination obtained, it is observed that the factor that most influences microwave heating is the chemistry of the materials, followed by mineralogy (closely associated with the chemistry of the rocks).

Table 10. Comparison of parameter T70 versus loss tangent.

	T70	ϵ''/ϵ'
Coefficient of determination for mineralogy	0.7963	0.5135
Coefficient of determination for chemistry	0.8633	0.6544
Necessary equipment	Microwave	Dielectric properties equipment
Sample preparation	Aggregate 4/8	Grinding
Results analysis	Direct	Require interpretation
Cost	Low	Expensive

Chemical elements such as Al_2O_3 and Fe_2O_3 positively affect the heating of aggregates for road construction (Table 9 and Equation 3). Other elements, i.e., MgO , TiO_2 , and MnO , seem to promote heating (despite their moderate-low R^2 values in Table 9). Nevertheless, their low content in stable aggregates for pavement construction and maintenance recommended not to consider them in the elaboration of Equation 3. On the other hand, chemical elements such as SiO_2 and CaO (in carbonate rocks) slow down the heating (Table 9 and Equation 3). Other authors have reported that part of these considerations on chemistry, chemical elements such as Fe_2O_3 [21,23,28,31] were considered one of the great promoters of microwave heating. However, this influence is low in silicate minerals, and Fe_2O_3 improves the heating performance when it is part of metallic minerals, impurities [12], or residues such as slag [30]. Other chemical elements such as MnO or TiO_2 were also presumed to be high microwave absorbers [21,28,32], but their influence on aggregates is low since they have little representativeness. Therefore, the literature considered other elements such as Al_2O_3 and MgO as medium microwave absorbers [25,29]. However, both resulted in high microwave absorbers in silicate rocks in this study, reducing the heating time. Concerning CaO , it has always been considered a low microwave absorber [28,30,31] because it was only studied in carbonate minerals. However, when it is associated with Al_2O_3 , MgO , and Fe_2O_3 in silicate minerals, it does not slow down the heating, so

together with these compounds, it is a harmless element **for** microwave applications. Finally, the elements of SiO₂ and CaO (in carbonate minerals) are considered low absorption, described in the literature by other authors [28,30,31]. The rest of the elements, such as CaO (in silicate minerals), K₂O, and Na₂O, do not affect mineral heating, although previously, Na₂O was considered a medium absorber [28,32].

Minerals that promote heating with microwaves are pyroxene, chlorite, biotite, and hornblende, these contain significant concentrations of heating-promoting chemicals such as Al₂O₃, Fe₂O₃, and MgO, and some of them contain water in their mineral structure [21] (Table 11). Some of these minerals, such as biotite and pyroxene, had been reported as minerals that favour mineral heating with microwaves [12,16,21], but the causes of this rapid heating had not been reported. In contrast, minerals such as quartz, calcite, and muscovite are considered weak microwave absorbers and slow down heating [12,16,21]. Other minerals such as plagioclase or feldspar are considered inert at the rate of heating.

Table 11. Chemical composition of minerals with high microwave response.

Mineral	Mineral formula
Pyroxene	(Ca,Mg,Fe) ₂ (Si,Al) ₂ O ₆
Chlorite	(Mg,Fe) ₃ (Si,Al) ₄ O ₁₀ (OH) ₂ ·(Mg,Fe) ₃ (OH) ₆
Biotite	K(Mg,Fe) ₃ AlSi ₃ O ₁₀ (OH, F) ₂
Hornblende	Ca ₂ (Mg,Fe,Al) ₅ (Al,Si) ₈ O ₂₂ (OH) ₂

Regarding the apparent density of the material and microwave heating, no relationship has been found. This may be due to the lack of correlation between both parameters or to discrepancies between other studies due to the use of different test methods or the type of samples used.

About the size of the crystals, moderate correlations have been obtained for quartz and calcite. The increase **in the size of** these minerals (minerals inert or with low susceptibility to microwaves) would produce slower heating. An increase in size can cause the microwave waves to be distributed more inefficiently and, therefore, the heating to be slower. This study has also demonstrated that two quartzite rocks (QRT-1 and QRT-2) with the same composition but different crystal sizes, QRT-1 fine grain and QRT-2 coarse grain, present different heating rates (higher for the QRT-2). In both cases, the heating rate is slow since they only consist of quartz, a mineral that has already been

considered inefficient but presents a slower heating rate when presenting a larger size [24].

It is possible that minerals that present rapid heating, such as pyroxene or chlorite, have faster heating when they present large crystal sizes (for example, poikilotopic textures). This can be intuited in the OPH-1 and OPH-4 samples, although it has not been statistically demonstrated due to the influence of other minerals. In the case of minerals with high heating rates and large crystals (poikilotopic texture), the distribution of microwave waves is homogeneous, and this is the main factor to improve the homogeneous heating in the rock [35,36].

These results will have a significant impact on the search for aggregates that present high microwave heating for self-healing applications [6,7], heating of aggregates for industry [41], and mining [27]. It will also save economic costs derived from maintenance or energy expenses [13] and environmental costs caused by CO₂ due to the burning of fossil fuels [5,13].

5. Conclusions

This research investigates the factors that affect the differential heating or susceptibility to microwaves of aggregates for road pavements. Different laboratory tests have studied the chemical properties (mineralogy and chemical) and physical properties (crystal size and apparent density) of the aggregates.

The dielectric permittivity (loss tangent) and the direct heating values (parameter T70) of the 24 aggregates studied have been correlated with each of the properties analysed. Predictive models of heating from elemental composition and mineralogy have been developed using linear and multiple correlation diagrams.

After analysing the results of this study, it is proved that the type and origin of the materials strongly affect the microwave heating process, obtaining the following conclusions:

- Mineral of high susceptibility favouring heating is **especially** pyroxene. Chlorite, biotite, and hornblende probably promote microwave heating, although additional research is needed to confirm this influence. On the other hand, minerals with low microwave susceptibility are quartz and muscovite. Since calcite is present at high contents in carbonatic rocks, it is not a good predictor of microwave susceptibility in this type of aggregates.
- The content of Al_2O_3 and Fe_2O_3 affects heating positively. The elements MgO , TiO_2 , and MnO , seem to promote heating, but their content in pavement aggregates are not representative. Elements such as SiO_2 and CaO (in carbonate rocks) negatively affect heating. Other chemical elements such as Na_2O , K_2O , and CaO (in silicate minerals) can be considered inert in microwave heating.
- Regarding the physical properties, the study of the size of the crystals was inconclusive due to the interference of multiple minerals. Nevertheless, in the case of mono-mineral rocks with low heating rate minerals, as the size of the crystals increases, the microwave heating gets slower.
- No relationship was found between the density data of the aggregates under study and their microwave heating data, possibly due to the low

1 porosity of the aggregates for pavements, the absence of water in the pores,
2 or a discrepancy between other studies in the test method or the samples
3 used.
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6 As a result, the chemical and mineralogical properties of the material are closely
7 related to differential microwave heating. Multiple regression models have been
8 elaborated to deduce these relationships, obtaining a strong correlation for the
9 chemistry and mineralogy of the rock versus the T70 parameter. However, the
10 regression models versus the dielectric loss tangent were weaker. Therefore, it is
11 recommended to apply the T70 prediction models to estimate the microwave
12 susceptibility of aggregates.
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20 The novelty of this paper is the study of the heating of a large number of aggregates
21 samples corresponding to different lithologies. In addition, knowledge has been
22 obtained on the natural factors (chemical and physical) that affect the heating of
23 aggregates and cause the differential heating of the different lithologies. This new
24 information helps dry aggregates facilities, on site de-icing of roads, self-repair
25 techniques of asphalt mixtures, and other applications to aggregates or pavements that
26 can be implemented with microwaves.
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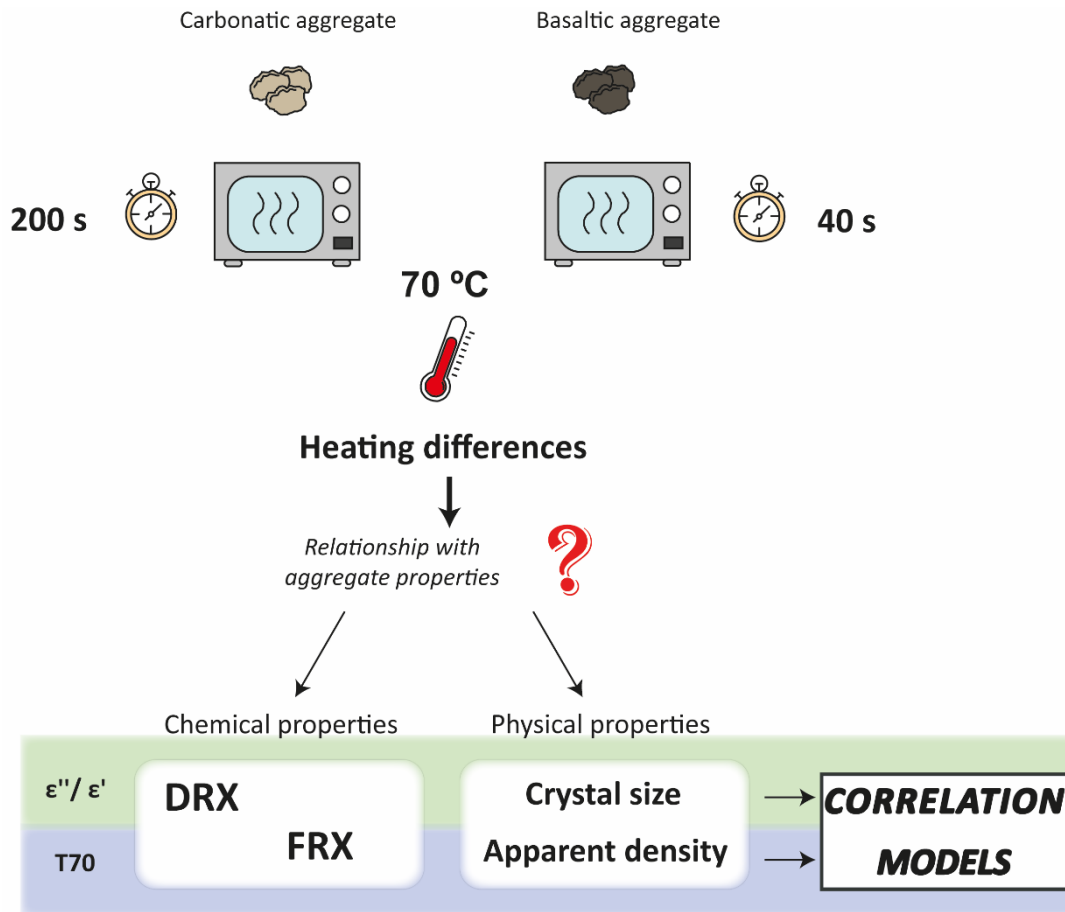
Natural factors related to the differential heating of aggregates exposed to microwaves

Keywords

Aggregates. Microwave heating. Petrography. Mineralogy. Chemical composition. Asphalt mixtures. Self-healing.

Highlights

- The chemical and mineralogical composition of aggregates affects microwave heating.
- Models have been proposed to predict microwave heating of aggregates for asphalt pavements.
- A relationship has been established between crystal size and heating in mono-mineral rocks.



Abstract

The heating produced by microwaves has been demonstrated to be effective in materials such as aggregates, although notable differences in heating have been observed between different types of aggregates. The geological factors of each material are responsible for these differences in heating due to compositional elements such as the mineralogy and chemistry of the material and physical characteristics such as crystal size and apparent density.

The study of each of these characteristics could unravel the factors determining differential heating in aggregates, which would be relevant for different applications in the mining industry (heating, drying, and comminution) and in civil engineering in self-healing (microcrack repair) or winter road maintenance (surface de-icing).

This paper investigates the influence of microwave heating on the chemical, mineralogical, and physical properties of aggregates susceptible to be used in asphalt mixtures. For this, aggregates with different characteristics have been selected. Various

1 tests were conducted to determine the factors affecting heating: thin-section studies
2 (mineralogy and crystal size), XRD (mineralogy) and XRF (chemistry) composition
3 studies, and bulk density of the rocks. These results were correlated with dielectric
4 permittivity data (loss tangent) and direct heating data (parameter T70). Both
5 techniques have proven helpful as an indicator of susceptibility to microwaves. Finally,
6 multiple regression models were proposed to apply this knowledge to any rocks suitable
7 as aggregate.
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16 1. Introduction

18 Aggregates are essential materials for society and are in high demand [1] for
19 different applications such as the manufacture of concrete and mortars, aggregates for
20 pavements, breakwaters, ballast, and other applications [2]. The sources of aggregates
21 are varied; for example, in Spain, the quarries of crushed stone stand out with 69.8%,
22 followed by sand and gravel pits with 25.7%, although there are by-products from other
23 mining operations 2.7%, and a growing but still low (1.9%) use of recycled and artificial
24 materials [2], which in some cases are also pre-treated to improve their properties [3,4].
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32 One of the highest aggregates consumption activities is road construction, using
33 approximately 28% of the aggregates. In addition to aggregates, asphalt mixtures
34 include other materials such as bituminous compounds, a primary source of carbon
35 emissions [5]. The current interest in climate change and the reduction of carbon
36 emissions have motivated recent research on the preventive maintenance of
37 pavements. One promising method is heating asphalt pavements by microwaves until
38 the bituminous material flows, producing the healing of cracks in a process known as
39 self-healing [6,7]. The studies carried out in the laboratory confirmed this technique as
40 an effective tool to extend the life span of asphalt pavements.
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50 Microwave heating is a green technology that demonstrates many advantages
51 over conventional heating methods. Microwave energy is rapidly transformed into heat
52 within the material through electromagnetic radiation. In contrast, in conventional
53 heating, thermal energy has to cross through the volume of the sample from the outside
54 [8]. Microwave heating could be considered a clean process since it uses electrical
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energy compared to other processes where heat is generated through combustion [9]. The advantages of this type of heating are rapid and contactless heating, selective heating of the material, volumetric heating (from inside of the material), and a high level of safety for operators [10–12], besides environmental advantages such as the reduction of atmospheric emissions [13]. Microwave applications in mineral processing have focused on assisted drying and crushing. Still, recently, other applications have emerged, such as mineral and rock heating, for applications related to the road industry, such as material heating to reduce manufacturing costs [13] or self-healing [14].

Microwave frequencies cover a part of the electromagnetic spectrum that extends from 300 MHz to 300 GHz [15]. The dielectric loss tangent defines the ability of a material to heat up in the presence of a microwave field:

$$\operatorname{tg} \delta = \frac{\varepsilon''}{\varepsilon'} \quad (1)$$

The dielectric loss tangent comprises two parameters, the dielectric constant (ε') and the dielectric loss factor (ε''). The dielectric constant or real permittivity determines the amount of incident energy not absorbed by the material. In contrast, the dielectric loss factor or imaginary permittivity measures the dissipation of electrical energy inside the material in the form of heat [10–12].

The factors that could influence heating, in addition to the real and imaginary permittivity (that depend on the frequency), are thermal conductivity, heat capacity, temperature, apparent density, sample size, particle size, presence of susceptors, chemical composition, microwave cavity geometry, and power level [15]. In addition, water content is often a contributing factor since water has a high loss factor [16].

Studies on the interaction of microwaves with minerals and rocks are scarce. Some works have attempted to classify rocks and minerals according to their responses to microwave radiation (rate of heating and maximum temperature) at different power levels. Still, the diversity of the studied rocks is very limited. One of the pilot studies related to civil engineering was carried out by Benedetto & Calvi [13], who tested two types of rocks (basalt and limestone), obtaining good heating results for basaltic

aggregates. Another study, which relates heating to mineralogy, is that of Garcia-Baños *et al.* [17], where four types of rock were studied (basalt, granite, sandstone, and marble), obtaining results similar to those of [13]. A study carried out by Li *et al.* [18] classifies rock heating by its origin, obtaining good results with higher heating for igneous rocks with basic composition than heating in igneous rocks of acid composition. However, sedimentary rocks presented a similar behaviour to igneous rocks of acid composition. Finally, the most complete studies in this area are those of Trigos *et al.* [14,19], in which various types of aggregates corresponding to blast furnace slag, carbonate (limestone and dolomite), volcanic (ophite, andesite, and basalt), plutonic (granite and granodiorite), and metamorphic rocks of different grades were studied. The results obtained from heating suggested that the composition of the rocks plays a key role in their differential heating.

Other studies focused on the behaviour of minerals instead of rocks. The first study is Chen *et al.* [20], which measured the heating of 40 minerals belonging to different groups. This work determined that the minerals that present more heating are sulphides and oxides with a metallic appearance; in contrast, minerals of the group of carbonates and silicates have low heating rates. Additional studies on the heating of sulphides obtained similar conclusions [18,21]. Although some studies showed that rock-forming minerals (silicates and carbonates) are weak microwave absorbers (absorbing microwave energy transforming it into heat), they are not completely inert to microwave radiation. The information on these minerals from different authors and their relationship to microwaves is consistent and allows the minerals to be classified according to their microwave absorption capacity. Prevalent minerals considered weak microwave absorbers are quartz, k-feldspar, plagioclase, and calcite [12,13,16,21–26]. Other common minerals, such as muscovite and hornblende, are considered medium absorbers [12,16,21,23]. Finally, biotite and pyroxene (enstatite) are good absorbers [12,23,26,27]. Other less common minerals are olivine and garnet (almandine), which behave as weak microwave absorbers [23]. It should be noted that microwave absorption and response can be conditioned by the origin, type, and content of impurities [12].

1 The influence of the chemical composition in the microwave heating process has
2 been studied in some works. The data collected from different authors allowed us to
3 differentiate three groups: high absorption (Fe_2O_3 , MnO , and TiO_2) [21,23,26,28–33],
4 medium absorption (Al_2O_3 , MgO , and Na_2O) [28,30–33], and low absorption (SiO_2 , K_2O ,
5 and CaO) [28,30,31]. Other compounds that facilitate heating even in trace
6 concentrations, and have high absorption, are NiO_2 , CoO , CuO , WO_3 , and CrO_3 [21,31].
7 The effect of elemental substitution and impurities can impact the permittivity of
8 minerals. Iron, chromium, and manganese oxides have higher permittivities than other
9 oxides [21].

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18 Another factor to consider is the crystal size and rock texture, related to the
19 durability of the rocks [34]. The most relevant work on this topic in microwave heating
20 is Nicco *et al.* [24]. Minerals that efficiently absorb microwaves and have high dielectric
21 constants are known to facilitate heating. However, the spatial distribution of these
22 grains is crucial since the heterogeneity of rock materials is essential in the response of
23 rocks subjected to microwave irradiation [12,35]. Therefore, heterogeneous properties
24 produce non-homogeneous thermal distribution and selective absorption between
25 minerals [24]. Fine-grained rocks can be heated more efficiently due to an adequate
26 distribution between the crystals since the heated minerals are closer [36,37]. On the
27 other hand, in coarse-grained rocks, the redistribution of heat is more inefficient,
28 causing the rock not to heat homogeneously and producing hot-spots [26,27].

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40 Some authors [33,38] have considered the apparent density factor in the
41 differences obtained in the dielectric permittivity. Other physical properties associated
42 with the density are the porosity [39] and water content [21,40] that can affect
43 permittivity and heating. A general classification of the influence of water present in the
44 material informed about its permittivity and microwave response was proposed by [21].
45 This classification is sorted from weakest to strongest: bulk water, surface, weakly
46 adsorbed, strongly adsorbed, hydrated, and hydroxyl.

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This study aims to know the chemical or physical properties that influence microwave heating of aggregates that give rise to differential heating for industrial and construction applications.

Several emerging technologies can benefit from this type of heating regarding the practical application of microwave radiation in asphalt materials and pavements. For example, some of the related microwave applications in the aggregates industry are material drying [41], crushing or industrial heating [42,43]. Other studies, already directly related to asphalt pavements, are for manufacturing asphalt mixtures, replacing the combustion of fossil fuels [13]. Likewise, prototypes have been proposed for the de-icing of the pavement in winter, based on the projection of radiation on the pavement [30]. These types of devices, already existing, allow heating pavement sections of the order of 1 m² to perform localised conservation operations [44] that could be applied on a larger scale, for in situ recycling of asphalt pavements or to promote the assisted repair of the most superficial layers of the pavement [45].

Regarding the repair of pavements, it must be taken into account that the asphalt mixture is made up of a combination of aggregates, additives, and bitumen, the aggregate being the main constituent with more than 90% by weight of the mixture, so the aggregates form an essential part of the asphalt mix. Furthermore, regarding the interaction of each of these components with microwaves, it is known that microwave heating is strongly influenced by the electromagnetic characteristics of the irradiated material, improving the efficiency of the system. The heating of the pavement is produced by the heating of the aggregates and the additives that compose it [7] since bitumen is transparent to microwaves [46]. Therefore, the heating of the asphalt pavement depends mainly on the aggregates to absorb the energy and transfer it to the asphalt.

Therefore, the results of this research may apply to these technologies in the construction and maintenance of roads. Furthermore, it should be considered that the electrical energy used by microwave devices, when it comes from renewable sources, notably improves the sustainability of these tasks, compared to traditional construction and conservation procedures, based on the consumption of fossil fuels. Hence the final objective of this research is the proposal of aggregates that optimise the energy consumption of these emerging technologies due to their response to microwave energy.

In order to know which aggregates have the best susceptibility properties (microwave heating), an experimental study was carried out on a broad spectrum of samples to determine the influence of the chemical and physical characteristics of the rocks in their response to heating in the face of the microwave exposure. A total of 24 samples were studied, some with similar compositions but different textures. A detailed analysis of the chemical properties (mineralogy and chemistry) and the physical properties (crystal size and apparent density) of the rocks under study has been carried out. In addition, microscopic observations were made to determine whether the warming causes are controlled by mineralogy, texture, or grain size. In addition, knowledge is offered on the influence of bulk density and grain size and the content of some chemical compounds such as iron, hydrated minerals, and their relationship between the dielectric properties of the rock.

Material characterisation is important in determining the applicability of microwaves to different rock types and optimising assisted processes by predicting materials that lead to efficient heating processes. The long-term objective is to provide knowledge that can propose rock types for industrial or construction techniques to make them more profitable from an economic and environmental point of view.

2. Materials and Methods

2.1. Materials

Twenty four aggregates of different compositions and origins have been studied (Table 1 and Figure 1). According to their composition, the rocks have been grouped into two categories: **a) Silicate aggregates**, ordered from highest to lowest SiO₂ content. Low SiO₂ content (basaltic rocks), medium SiO₂ content (schist and granitic rocks), and high SiO₂ content (quartzites), and **b) Carbonate aggregates** (limestone, dolostone and marble). The rocks have been classified according to their composition and origin.

SAMPLES	CATEGORY	SAMPLES	CATEGORY
OPH-1	Silicate aggregate	SCH-1	Silicate aggregate
OPH-2	Silicate aggregate	MYT-1	Silicate aggregate
OPH-3	Silicate aggregate	GRB-1	Silicate aggregate

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OPH-4	Silicate aggregate	GRA-1	Silicate aggregate
OPH-5	Silicate aggregate	GRA-2	Silicate aggregate
AND-1	Silicate aggregate	QRT-1	Silicate aggregate
AND-2	Silicate aggregate	QRT-2	Silicate aggregate
BAS-1	Silicate aggregate	CAL-1	Carbonate aggregate
BAS-2	Silicate aggregate	CAL-2	Carbonate aggregate
MSD-1	Silicate aggregate	CAL-3	Carbonate aggregate
PHI-1	Silicate aggregate	DOL-1	Carbonate aggregate
AMP-1	Silicate aggregate	MAR-1	Carbonate aggregate

Table 1.
Aggregates under
microwave study.



Fig. 1: Samples of 24 aggregates under study.

2.2. Physical characterisation, petrographic analysis, and compositional description

Several tests were carried out to study the physical properties and petrographic, mineralogical, and chemical characteristics. First, the apparent density, described in the standard EN 1936: 2007, was measured [47].

For the petrographic study (textural and compositional description of the rock), the authors followed EN 12407 standard (Natural stone test methods, Petrographic examination) [48]. The terms used in the description are included in the EN 12670 standard (Natural stone. Terminology) [49]. A Zeiss West Germany Optical 316 Microscope with 2.5x, 10x, and 20x magnifications were used for microscopic characterisation with transmitted and polarised light sources. Thin sections, 30 μm -thick, of each rock type were prepared to characterize the mineralogical components.

The point-counting technique has been used to estimate the percentage by volume of minerals forming each sample. It is one of the most common methods for rock classification, for establishing trends and knowing the relative proportions and size distribution of the rock components. Point counting has been made with the software JMicrovision® on pictures of thin sections made with a camera attached to the petrographic microscope. After point counting analysis, measurements of the size (area) and shape of the minerals were made to obtain information on the textural relations and the size of the rock crystals (Fig.2). Finally, a validation of the data was carried out, eliminating the extreme values, and the homogeneity and normality of the data were checked.

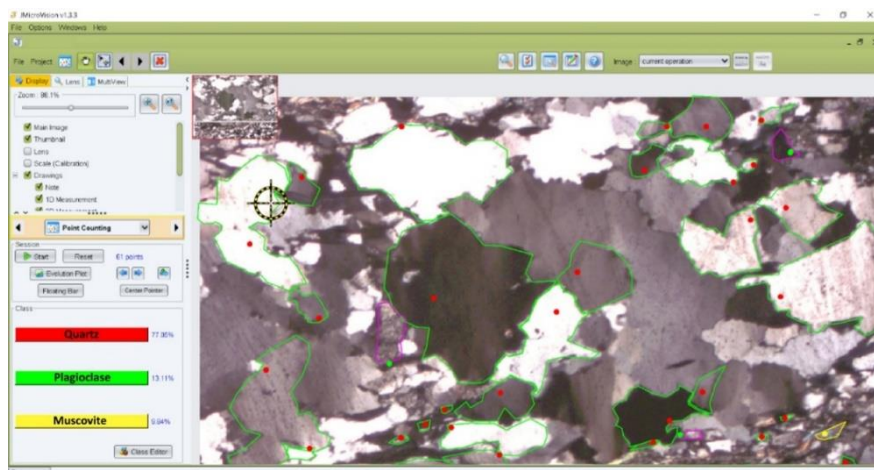


Fig. 2: Sample analysed by JMicrovision®.

1 Bulk mineralogy was determined by X-ray diffraction (XRD). A portion of the
2 samples was ground in an agate mortar to obtain a 53 μm powder. The non-oriented
3 powder was examined with a Philips PW 1720 diffractometer with Cu ($K\alpha$) radiation and
4 a Bruker D8 Advance diffractometer equipped with a Sol-X detector and Cu ($K\alpha$)
5 radiation in the CAI of Geological Techniques (Complutense University of Madrid). The
6 quantification of mineral phases was carried out using the Chung method [50] by Bruker
7 software (EVA[®]). Finally, X-ray fluorescence (XRF) was performed using a wavelength
8 dispersive X-ray fluorescence spectrometer (Bruker, S2 Ranger) to obtain a semi-
9 quantitative analysis of the chemical composition of the sample.
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23 **2.3. Microwave heating**

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27 To study the heating produced by microwave energy in different aggregates, the
28 authors used the T70 protocol proposed in [19], based on the direct measurement of
29 heating in an aggregate sample subjected to microwave radiation. The test measures
30 the time in seconds that a sample of 200 g of aggregate requires to reach 70 °C, from an
31 ambient temperature of 20 °C, by microwave radiation with an output power of 700 W
32 and a frequency of 2.45 GHz.
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40 For this test, the fraction 4/8 mm was used to avoid potential heating differences
41 related to the size of the particles. Before performing the microwave heating, the
42 aggregates were washed and dried in an oven at a temperature slightly below 100 °C.
43 Once the aggregate got dried and reached room temperature again (20 °C), a sample of
44 200g was introduced in a polypropylene plastic container that allows the heating of the
45 aggregate (polypropylene is utterly transparent to microwaves).
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1 An Orbegozo MI-2014 20L microwave oven was used, with an output power of
2 700 W and a frequency of 2.45 GHz (Figure 3); the maximum power was used during
3 the tests. During the T70 test, the temperature of the aggregate surface was measured
4 with an infrared temperature gun (Testo 830-T1), heating the aggregate at fixed time
5 intervals (every 20 seconds) until the bulk aggregate reached a surface temperature
6 above 110 °C.
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31 **Fig. 3:** Equipment used for the heating test.
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36 During the design of the experiment, the randomisation principle was followed
37 with random number tables to ensure the independence of the data. Furthermore, the
38 experiment was repeated 25 times per sample to avoid underestimating the variability.
39 During this process, measurements of the ambient temperature were taken to
40 determine its impact on the results. Subsequently, the data diagnosis was checked to
41 ensure correct (homoscedasticity, normality, and independence). Finally, the average,
42 range and standard deviation were calculated.
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58 **2.4. Permittivity measurements**

59 The dielectric properties of the sample have been determined by the dual-mode
60 microwave system, where the dielectric properties are calculated using a modified
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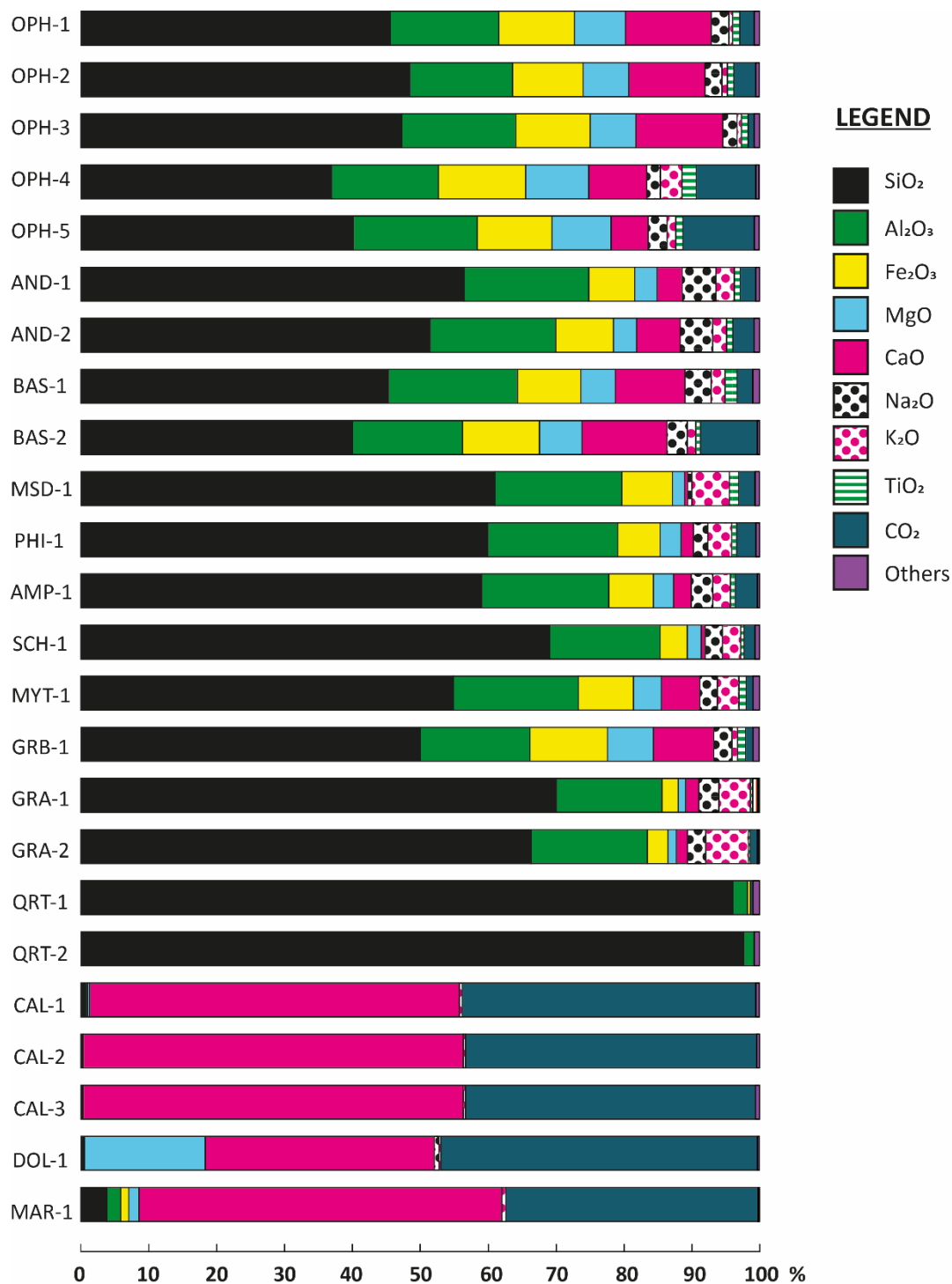


Fig. 4: Chemical data (XRF) for each sample.

Table 3. Chemical data (XRF) for each sample are given in percentage by weight.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	MnO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	SO ₃	Cr ₂ O ₃	CO ₂	Others
OPH-1	45.60	16.03	11.14	7.50	0.06	12.66	2.60	0.54	1.09		0.09	0.08	2.10	0.55
OPH-2	48.50	15.12	10.39	6.77	0.06	11.22	2.52	0.81	0.92		0.09		3.22	0.24
OPH-3	47.30	16.84	10.91	6.74	0.07	12.82	2.08	0.69	0.98		0.09		0.89	0.50
OPH-4	36.90	15.74	12.87	9.30	0.06	8.54	2.01	3.15	2.07	0.27	0.07		8.84	0.16
OPH-5	40.18	18.24	11.03	8.68	0.08	5.44	2.77	1.30	1.06	0.23	0.23		10.56	0.17
AND-1	56.38	18.41	6.78	3.30	0.05	3.70	5.00	2.63	0.86	0.13	0.08		2.30	0.45
AND-2	51.37	18.54	8.51	3.40	0.05	6.42	4.80	2.06	0.93	0.35	0.06		3.10	0.32
BAS-1	45.33	19.00	9.32	5.08	0.06	10.24	3.91	2.06	1.77	0.44	0.07		2.31	0.36
BAS-2	39.92	16.25	11.31	6.23	0.07	12.50	3.02	1.24	0.72	0.05	0.05		8.37	0.17
MSD-1	61.02	18.70	7.51	1.80	0.04	0.46	0.60	5.55	1.36		0.11		2.40	0.40
PHI-1	59.97	19.16	6.22	3.11	0.04	1.81	2.14	3.51	0.75		0.20		2.84	0.21
AMP-1	59.07	18.71	6.53	3.00	0.04	2.60	3.19	2.56	0.75		0.11		3.25	0.19
SCH-1	69.11	16.34	4.00	2.07	0.03	0.57	2.56	2.70	0.51		0.25		1.66	0.19
MYT-1	54.90	18.43	8.13	4.10	0.05	5.67	2.60	3.21	1.15	0.30	0.07		1.00	0.27
GRB-1	49.89	16.18	11.45	6.73	0.07	8.90	2.67	0.85	1.16	0.13	0.58		1.10	0.27
GRA-1	70.09	15.58	2.39	1.09	0.02	1.92	2.98	4.64	0.35		0.05		0.53	0.25
GRA-2	66.37	17.14	3.02	1.29	0.03	1.64	2.67	6.31	0.31				1.03	0.19
QRT-1	96.09	2.15	0.44	0.20		0.07	0.10	0.14	0.10		0.06		0.40	0.16
QRT-2	97.72	1.51	0.19					0.04	0.11		0.03		0.20	0.17
CAL-1	0.72	0.27	0.09	0.30		54.64		0.41			0.05		43.30	0.16
CAL-2	0.35	0.11	0.06	0.23		55.79	0.06	0.34			0.01		42.95	0.09
CAL-3	0.32	0.11	0.09	0.23		56.01	0.01	0.37		0.01	0.01		42.76	0.09
DOL-1	0.37		0.17	17.80	0.01	33.74	0.70	0.27					46.70	0.28
MAR-1	3.78	2.04	1.17	1.51	0.07	53.37	0.06	0.60	0.08		0.03		37.19	0.16

Table 4. Apparent density values in g/cm³.

SAMPLE	PHYSICAL PROPERTIES	SAMPLE	PHYSICAL PROPERTIES
	Apparent density		Apparent density
OPH-1	2.832	SCH-1	2.620
OPH-2	2.784	MYT-1	2.773
OPH-3	2.988	GRB-1	2.758
OPH-4	2.979	GRA-1	2.671
OPH-5	2.877	GRA-2	2.746
AND-1	2.676	QRT-1	2.696
AND-2	2.781	QRT-2	2.713
BAS-1	2.905	CAL-1	2.629
BAS-2	2.873	CAL-2	2.609
MSD-1	2.750	CAL-3	2.650
PHI-1	2.747	DOL-1	2.800
AMP-1	2.743	MAR-1	2.864

Based on the petrographic, mineralogical, and chemical features of the rocks, a classification of the samples has been established. For silicate rocks, two different classifications have been used depending on the genetic origin of the rock. The QAPF diagram of Le Maitre [52] was used to classify igneous rocks according to their modal composition, and the Eskola method modified by Turner and Verhoogen [53] was used for metamorphic rocks. Finally, two classifications have been used for the classification of carbonate rocks: Dunham [54] for rocks of calcitic composition and Friedman [55] for rocks with dolomitic composition.

To facilitate the description of the samples, they were grouped into five categories according to their characteristics, such as mineralogy, chemistry, or texture (Fig. 5). This description will also include the physical characteristics (apparent density and loss tangent).

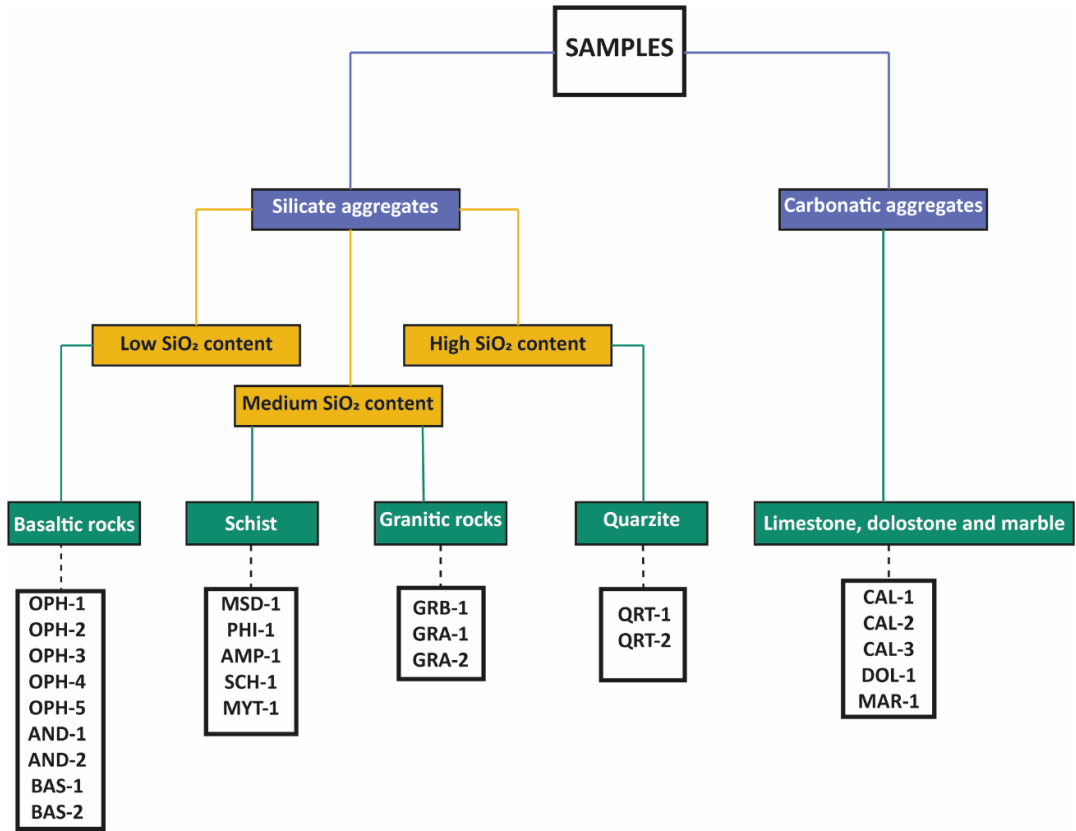


Fig. 5: Samples were studied and their classification according to their composition.

1 A) Silicate aggregates, basaltic rocks (OPH-1, OPH-2, OPH-3, OPH-4, OPH-5,
2 AND-1, AND-2, BAS-1, and BAS-2):
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4 The rocks in this group are volcanic rocks of basic composition with aphanitic or
5 porphyritic textures. The OPH-1, OPH-2, OPH-3, OPH-4, and OPH-5 correspond to
6 diabbases/ophites with plagioclase, augite, and hornblende. In OPH-1 and OPH-3
7 samples, augite minerals form large poikilotopic crystals, encapsulating smaller
8 plagioclase crystals (ophites). The OPH-2, OPH-4, and OPH-5 samples are similar in
9 composition to the previous ones, but the augite crystals do not form poikilotopic
10 crystals (diabbases), both rock groups equigranular texture with a fine-grained
11 intergranular tendency.
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20 XRD results show that the most abundant phases are plagioclase, chlorite,
21 pyroxene (augite), and hornblende. Other minor components are biotite, muscovite,
22 calcite/dolomite, and quartz. The two last minerals correspond to cement from a later
23 stage of rock formation. XRF results show values of SiO₂ between 45-40%, Al₂O₃ 17%,
24 Fe₂O₃ 12%, MgO 7-9%, CaO 12-6%, and TiO₂ 1-2%. The values of CO₂ are low except in
25 the samples OPH-4 and OPH-5 due to the presence of 9% of carbonatic cement.
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33 Finally, the density of the samples ranged between 2.8 -3.0 g/cm³, with higher
34 values in OPH-4 and OPH-5. The loss tangent value at 25 °C for the OPH-1, OPH-2, OPH-
35 3, and OPH-5 samples are 0.015, and for the OPH-4 sample, it is near 0.03.
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40 The samples AND-1 and AND-2 correspond to andesite, with a fine-medium grain
41 hypocrySTALLINE texture. The predominant mineral phases correspond to plagioclase,
42 hornblende, and volcanic glass. Finally, the BAS-1 and BAS-2 samples correspond to
43 basalts with aphanitic (microcrystalline) texture. The predominant phases are anorthite,
44 amphibole, chlorite, and olivine.
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50 XRD results for andesite and basalt samples are very similar. The most abundant
51 phases are plagioclase with values between 30-50%, hornblende >30%, chlorite, olivine
52 (only in basalt), biotite, quartz (volcanic glass), and calcite cement. The XRF results show
53 variable percentages of the different components with 40-54% SiO₂ values, higher in the
54 andesites, Al₂O₃ 16-19%, Fe₂O₃ 7-11%, MgO 3-6%, CaO 4-13%, Na₂O 3-5%, K₂O 2%, and
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1 TiO₂ 1%. The BAS-1 and BAS-2 samples present a higher CaO value due to the carbonate
2 cement formation formed during a hydrothermal process.
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4 Finally, the density is 2.6-2.7 g/cm³ for andesite and 2.8-2.9 g/cm³ for the basalt
5 samples. The loss tangent at 25 °C ranges from 0.028-0.022 for the andesite samples
6 and 0.017 for the basalt samples.
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16 *B) Silicate aggregates, schist rocks (MSD-1, PHI-1, AMP-1, SCH-1, and MYT-1):*
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18 The rocks of this group are metamorphic with a foliated texture and a silica
19 content ranging from 50% to 70%. Sample SCH-1 corresponds to schist, and MSD-1
20 corresponds to a combination of schist and andesite. Both have a lepidoblastic texture
21 of medium grain, and the predominant minerals are quartz and minerals from the mica
22 group. The PHI-1 sample corresponds to phyllite with a fine-grained lepidoblastic
23 texture, and the predominant minerals are quartz and minerals from the mica group
24 (biotite and chlorite). The sample MYT-1 corresponds to mylonite, a rock formed by
25 dynamic metamorphism, with a fine-grained granular texture, a high content of quartz,
26 and a lower content of other minerals such as plagioclase and muscovite. Finally, the
27 AMP-1 sample corresponds to a fine-grained amphibolite with decused texture and
28 abundant chlorite due to hydrothermal activity.
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41 The results obtained for the mineralogy (XRD) of all samples of this group show
42 that they have the typical mineralogy for these lithologies, with variable values of quartz,
43 plagioclase, muscovite, and chlorite. The XRF results show that the most abundant
44 element is SiO₂ with values between 55-70%; the rest of the elements are similar
45 between the different aggregates with approximate values of Al₂O₃ 17%, Fe₂O₃ 7%, MgO
46 3%, K₂O 3%, and Na₂O, and TiO₂ values between 0.5-1.5%.
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53 The density of these samples is 2.7 g/cm³, and the loss tangent value at 25 °C is
54 less than 0.01 for SCH-1, PHI-1, AMP-1, AMP-1, and while for MSD-1, the value is 0.0141.
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C) Silicate aggregates, granitic rocks (GBR-1, GRA-1, and GRA-2):

The rocks of this group are plutonic rocks of acidic or intermediate composition. The samples GRA-1 and GRA-2 correspond to granites with a similar composition, predominantly quartz, feldspar, and biotite with a granite texture of the medium-fine grain. The GRB-1 sample corresponds to diorite. The predominant minerals in GRB-1 are quartz, hornblende, and plagioclase, with a granite texture of the medium-fine grain.

XRD results show that the GRB-1 rock has approximately 30% quartz, while 55% of GRA-1 and GRA-2 samples are quartz. Other differences between the samples are 10-20% K-feldspar in the GRA-1 and GRA-2 samples and the presence of 30% hornblende in GRB-1. The rest of the phases are similar in all samples: plagioclase, biotite, and chlorite. The FRX results show significant differences in some elements between the rocks GRB-1, GRA-1, and GRA-2, the content of silica is 70% in the case of granites and 50% for diorite. In the GRB-1, percentages of Fe₂O₃ are 12%, Al₂O₃ 16%, MgO 7%, CaO 9%, and TiO₂ 1%. In the case of GRA-1 and GRA-2 are Al₂O₃ 16%, Fe₂O₃ 3%, MgO 1%, CaO 2%, K₂O 5-6%, and TiO₂ <0.5%.

Finally, the apparent density of the samples is 2.7 g/cm³, and the value of the loss tangent is less than 0.01 for GRB-1 and GRA-1, but the sample GRB-2 value is 0.0152 at 25 °C.

D) Silicate aggregates, quartzites (QRT-1 and QRT-2):

Non-foliated metamorphic rocks (granular texture) with a high silica content form this group, corresponding to quartzites with a quartz content above 95%. The difference between both samples is the crystal size: QRT-1 has fine-medium size crystals, while the crystals in QRT-2 are coarser.

XRD results show that the only mineral phase present is quartz. The XRF results for both samples are similar, with the content of SiO₂ greater than 96% and about 2% of Al₂O₃.

The density of the samples is 2.7 g/cm³, and the value of the loss tangent is less than 0.005 at 25 °C.

E) *Carbonatic aggregates (CAL-1, CAL-2, CAL-3, DOL-1, and MAR-1):*

This group consists of sedimentary rocks of carbonatic composition, except the sample MAR-1 that is metamorphic. CAL-1 and CAL-2, 3 correspond to limestones with micritic texture and sparitic cement in porosity, CAL-3 correspond to limestones with grainstone texture and sparitic cement in porosity. Sample DOL-1 corresponds to a dolostone, and sample MAR-1 to a calcite and dolomite marble with a fine-grained crystalline texture.

XRD results show that the predominant mineral phases are calcite, dolomite, and traces of quartz. The FRX results show that the CaO values are around 55%, except in the case of dolomite, with 34% CaO and 17% MgO. In addition, the MAR-1 aggregate presents other components in its composition, such as SiO₂ 4%, Al₂O₃ 2%, Fe₂O₃ 1%, and MgO 1%.

Finally, the density of the limestone samples is 2.6 g/cm³, and for the samples of dolomite and marble, 2.8 g/cm³. Concerning the loss tangent, the values are below 0.01 at 25 °C.

3.2. Crystal size assessment

The measurement area of the minerals present in each aggregate has been collected. The values obtained for the crystal sizes are between 0.001 and 1.5 mm²; this allows us to observe the differences between the crystal sizes in each group of rocks. The minerals with a total content below 5% of the rock volume have been omitted.

Regarding the average crystal size, rocks can be classified into three groups: coarse, medium, and fine. The coarse-grained group comprises granites, quartzite (QRT-2), and marble, with mean values above 0.2 mm². The group of medium-grained rocks is formed by samples of andesite, mylonite, granodiorite, and quartzite (QRT-1), all with average values between 0.025 and 0.2 mm². Finally, the fine-grained rocks group consists of samples of ophite, basalt, meta-sandstone, phyllite, amphibolite, schist, carbonate, and dolomite, with values below 0.025 mm².

A detailed study has been carried out, differentiating the minerals of each rock and indicating in which rocks each mineral is found. To get this diagram, the measurement area of each mineral in different aggregates has been collected (Fig.6). The values obtained are between 0.001 and 1.5 mm² for the different samples studied to observe differences between minerals. A total of 11 minerals have been differentiated: biotite, hornblende, pyroxene, calcite/dolomite, K-feldspar, chlorite, olivine, muscovite, quartz, plagioclase, and iron oxides. The minerals have been compared to each other. A description of the results obtained for the minerals studied by crystal size has been made in Table 5.

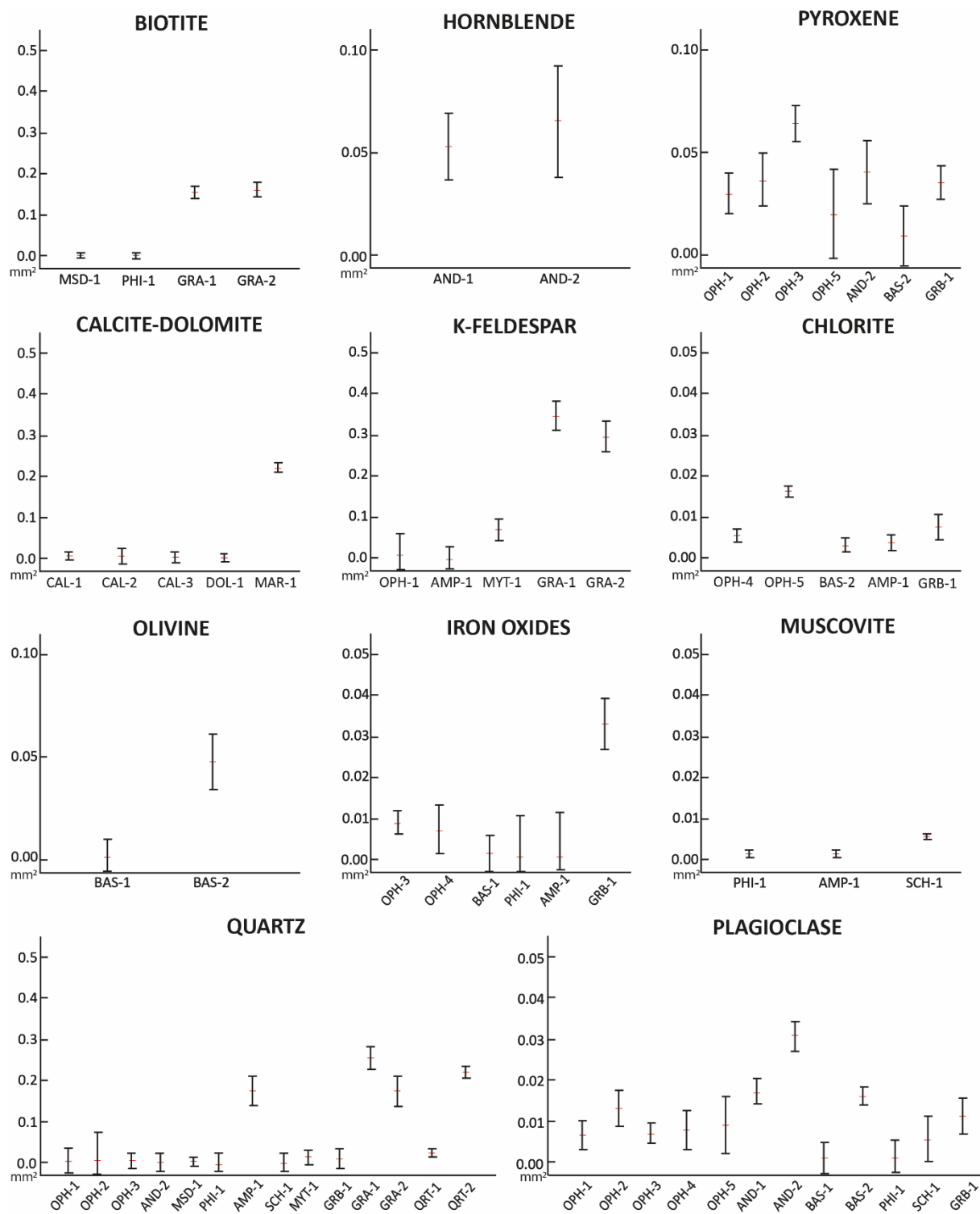


Fig. 6: Distribution of crystal size in different minerals.

Table 5. Summary of crystal size of minerals.

Mineral	Rock	Crystal size
Biotite	- Foliated (MSD-1 and PHI-1) - Granitic (GRA-1 and GRA-2)	Coarse grain: GRA-1 and GRA-2 Fine-grain: MSD-1 and PHI-1
Hornblende	Basaltic (AND-1 and AND-2)	Fine-grain: AND-1 and AND-2
Pyroxene	- Basaltic (OPH-1, OPH-2, OPH-3, OPH-5, AND-2, and BAS-2) - Granitic (GRB-1)	Medium-fine grain: OPH-1, OPH-2, OPH-3, OPH-5, AND-2, and GRB-1 Fine-grain: BAS-2
Calcite/Dolomite	Carbonatic (CAL-1, CAL-2, CAL-3, DOL-1, and MAR-1)	Coarse grain: MAR-1 Fine grain: CAL-1, CAL-2, CAL-3, and DOL-1
K-Feldspar	- Basaltic (OPH-1) - Foliated (AMP-1 and MYT-1) - Granitic (GRA-1 and GRA-2)	Coarse grain: GRA-1 and GRA-2 Fine-grain: OPH-1, AMP-1, and MYT-1
Chlorite	- Basaltic (OPH-4, OPH-5, and BAS-2) - Foliated (AMP-1) - Granitic (GRB-1)	Coarse grain: OPH-5 Medium-fine grain: OPH-4, BAS-2, AMP-1, and GRB-1
Olivine	Basaltic (BAS-1 and BAS-2)	Coarse grain BAS-1 Fine-grain: BAS-2
Muscovite	Foliated (PHI-1, AMP-1, and SCH-1)	Fine-grain: PHI-1, AMP-1, and SCH-1
Quartz	- Basaltic (OPH-1, OPH-2, OPH-3, and AND-2) - Foliated (MSD-1, PHI-1, AMP-1, SCH-1, and MYT-1) - Granitic (GRB-1, GRA-1, and GRA-2) - Quartzites (QRT-1 and QRT-2).	Coarse grain: QRT-2, GRA-1, and GRA-2 Fine grain: OPH-1, OPH-2, OPH-3, AND-2, MSD-1, PHI-1, AMP-1, SCH-1, MYT-1, GRB-1, and QRT-1
Plagioclase	- Basaltic (OPH-1, OPH-2, OPH-3, OPH-4, OPH-5, AND-1, AND-2, BAS-1, and BAS-2) - Foliated (PHI-1 and SCH-1) - Granitic (GRB-1).	Coarse grain: AND-2 Medium-fine grain: OPH-2, AND-1, and BAS-2 Fine-grain: OPH-1, OPH-3, OPH-4, OPH-5, BAS-1, PHI-1, SCH-1 and GRB-1
Iron oxides	- Basaltic (OPH-3, OPH-4, AND-2, and BAS-1) - Foliated (PHI-1 and AMP-1) - Granitic (GRB-1).	Medium grain: GRB-1 Fine-grain OPH-3, OPH-4, AND-2, BAS-1, PHI-1, and AMP-1

3.3. Analysis of microwave heating

The microwave heating results obtained have been used to calculate the value of T70 for each aggregate, classifying them into five classes. The first class corresponds to samples with faster heating: from 20 °C to 70 °C in less than 25 seconds. The second class includes samples that require between 25 and 50 seconds. The third class with samples that presented medium heating, passing from 20 °C to 70 °C between 50 and 100 seconds. The fourth class samples (slower heating) need between 100 and 200 seconds. Finally, the fifth class includes the samples that require more than 200 seconds to pass from 20 °C to 70 °C.

Table 6 shows the T70 values for each sample. The samples with high microwave susceptibility (HS), with T70 between 25 and 50s, are both andesites (AND-1 and AND-2). Samples between 50 and 100 seconds (medium susceptibility, MS) belong to multiple groups: subvolcanic rocks (OPH-1, OPH-2, OPH-3, OPH-4, and OPH-5), volcanic rocks, corresponding to basalts (BAS-1 and BAS-2); and the last group, foliated metamorphic rock with a sample (MSD -1). The T70 class samples between 100 and 200 seconds (low susceptibility, LS) belong to multiple groups: plutonic rocks (GRB-1, GRA-1, and GRA-2), metamorphic rocks (foliated) with different degrees (PHI- 1, AMP-1, and MYT-1) and sedimentary rock (DOL-1). Finally, the group of rocks that have a very low susceptibility (VLS) to microwaves (T70> 200s) are foliated metamorphic rocks (SCH-1), non-foliated metamorphic rocks: river-gravel quartzite (QRT-1), crushed quartzite (QRT-2), and marble (MAR-1).

Table 6. T70 values for samples.

SAMPLES	T70 (s)	SAMPLES	T70 (s)
OPH-1	57.7	SCH-1	245.4
OPH-2	97.2	MYT-1	153.4
OPH-3	75.1	GRB-1	113.9
OPH-4	65.8	GRA-1	161.8
OPH-5	78.2	GRA-2	118.8
AND-1	42.3	QRT-1	289.7
AND-2	47.4	QRT-2	504.2
BAS-1	72.2	CAL-1	334.5
BAS- 2	67.7	CAL-2	310.9
MSD-1	86.7	CAL-3	426.7
PHI-1	112.7	DOL-1	123.5
AMP-1	120.6	MAR-1	351.4

The temperature rise of the sample during microwave irradiation is linear with energy consumption [14], so the slope of the line represents the susceptibility of the material to microwaves. The steeper the slope of the line, the greater the susceptibility to microwaves and the more efficient the process. The calculated slopes are presented in Figure 7. It can be seen that the rocks of andesite, ophite, and basalt are the samples most susceptible to microwaves.

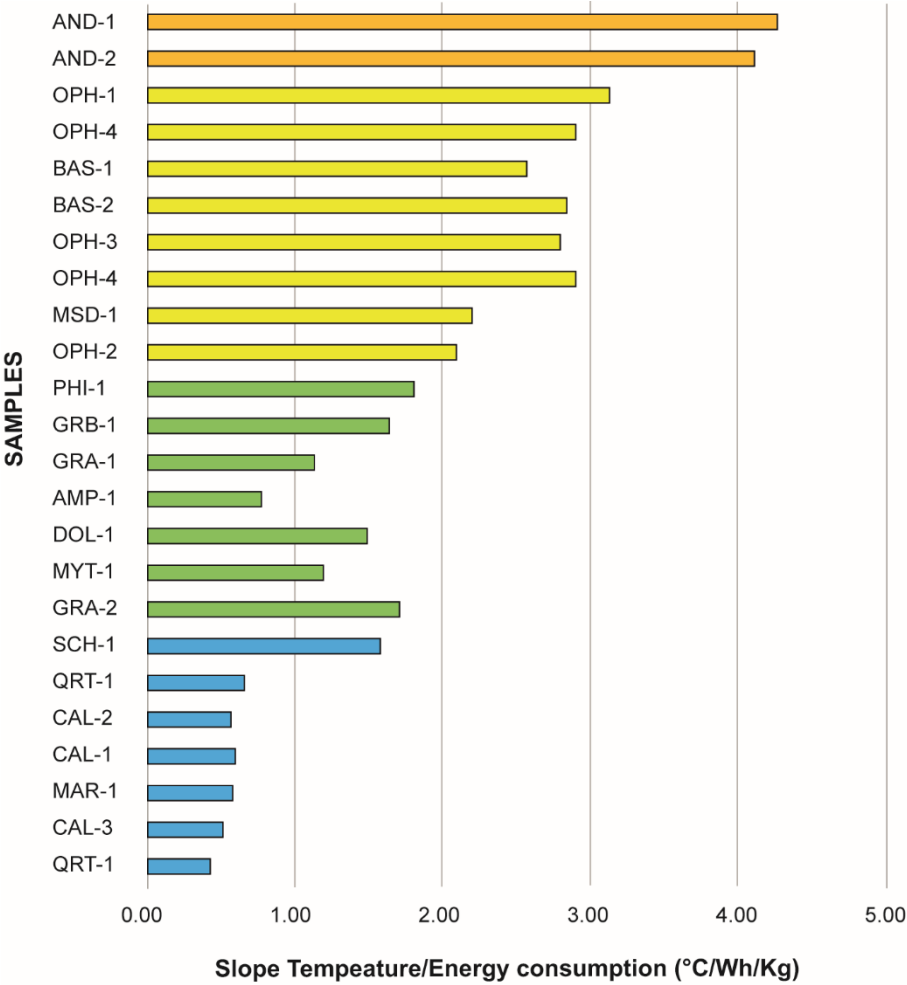


Fig.7: Slope of temperature versus energy consumption (susceptibility to microwaves). Samples were ordered from lowest to highest T70.

3.4. Results of the dielectric parameters

It has been reported that the dielectric permittivity values have a high relationship with the microwave susceptibility values of aggregates [19]. Therefore, in Table 7, the values of permittivity obtained in this study at 25 °C employing the CPM technique are shown. The values of the dielectric constant (ϵ'), loss factor (ϵ''), and loss tangent are included. The loss factor is calculated as the relationship between the loss factor and the dielectric constant (ϵ''/ϵ').

Table 7. Permittivity values.

SAMPLE	DIELECTRIC PROPERTIES		
	ϵ'	ϵ''	ϵ''/ϵ'
OPH-1	2.5949	0.0353	0.0136
OPH-2	3.0512	0.0463	0.0152
OPH-3	3.3398	0.0479	0.0143
OPH-4	2.7613	0.0804	0.0291
OPH-5	2.4578	0.0262	0.0107
AND-1	2.4380	0.0686	0.0281
AND-2	2.5275	0.0574	0.0227
BAS-1	3.3053	0.0562	0.0170
BAS-2	3.0203	0.0516	0.0171
MSD-1	2.6045	0.0366	0.0141
PHI-1	2.5752	0.0175	0.0068
AMP-1	2.7260	0.0144	0.0053
SCH-1	2.5756	0.0195	0.0076
MYT-1	2.5855	0.0160	0.0062
GRB-1	3.0580	0.0255	0.0083
GRA-1	2.1630	0.0132	0.0061
GRA-2	2.0679	0.0313	0.0152
QRT-1	1.9863	0.0061	0.0031
QRT-2	2.3947	0.0043	0.0018
CAL-1	3.3373	0.0132	0.0040
CAL-2	3.0184	0.0163	0.0054
CAL-3	3.2083	0.0141	0.0044
DOL-1	3.1244	0.0103	0.0033
MAR-1	3.0274	0.0237	0.0078

4. Discussion

Different studies have shown that the differential heating of materials with microwaves could be caused by various physical characteristics (crystal size and density) or compositional characteristics (mineralogy and chemistry). In the studies of [14,19], aggregates with different compositions were tested, and it was observed, using the parameter T70, the existence of differential heating among aggregates of different nature. The study [19] revealed that measurements of dielectric permittivity (loss tangent) provide results about the susceptibility to the microwave of aggregates similar to those obtained with the parameter T70, displaying a high correlation ($R^2 = 0.83$). To determine the factors influencing the heating response, the heating data (parameter T70) and dielectric permittivity (loss tangent) values should be compared with the values of mineralogy, chemistry, crystal size, and apparent density to observe the influence of these factors.

The minerals and elements detected in the XRD and XRF tests were compared with the dielectric permittivity (loss tangent) and microwave heating data (parameter T70). The resulting data were used for several regression models as described in the following sections.

4.1. Relationship of mineralogical, chemical, and physical properties with loss tangent and microwave heating (T70)

Mineralogical composition

From the results obtained by XRD, 17 different mineral phases (formation and alteration minerals) were obtained, only 11 mineral phases have been studied because only the formation minerals have been considered, eliminating alteration minerals and minerals with a low proportion (<5%). Silicate minerals including quartz, plagioclase, feldspar, biotite, muscovite, chlorite, hornblende, pyroxene, and olivine have been studied. The carbonate minerals included in the study were calcite and dolomite. Finally, olivine and dolomite minerals have been removed due to insufficient data.

Correlations between minerals and loss tangent are low (Table 8) due to the absence of correlation or the low presence of some minerals (olivine and dolomite). However, two strong correlations are observed for muscovite and calcite, with negative slope equations and high R^2 , indicating that a higher presence of these minerals reduces the loss tangent values as previously reported [23].

In the same line, the 11 most relevant mineral phases have been correlated with the parameter T70. Strong heating correlations have been found in the minerals studied with pyroxene and muscovite and moderate correlations in the case of quartz (Table 8). Summarising, based on the sign of the slope of the equation, muscovite and quartz reduced the heating, while pyroxene promoted the heating potential of microwaves. Quartz, muscovite, and pyroxene behaviour is consistent with the findings of other authors [12,21,26,56].

In other minerals, moderate to weak correlations have been obtained, and this may be due to different causes, such as concentrations of similar minerals between different rocks or low mineral content in the rock. Therefore, finding moderate correlations due to these causes does not indicate that the mineral influences microwave heating.

Table 8. Equations and Pearson's correlation coefficients for minerals versus loss tangent and T70 parameter.

Minerals	ϵ''/ϵ'		T70	
	Equation	R^2	Equation	R^2
Quartz	$y = -8E-05x + 0.0144$	0.16	$y = 2.7322x + 30.551$	0.53
Biotite	$y = -0.0004x + 0.0146$	0.03	$y = 7.8573x + 71.769$	0.27
Hornblende	$y = 0.0001x + 0.0153$	0.03	$y = -0.2543x + 78.986$	0.01
Plagioclase	$y = 0.0001x + 0.01$	0.07	$y = -0.2565x + 110.22$	0.01
Muscovite	$y = -0.0001x + 0.0142$	0.91	$y = 0.747x + 74.395$	0.79
K-feldspar	$y = 0.0006x + 0.0058$	0.24	$y = -2.2314x + 156.3$	0.20
Chlorite	$y = 0.0002x + 0.01$	0.17	$y = -2.3553x + 137.38$	0.29
Pyroxene	$y = -8E-05x + 0.0146$	0.09	$y = -1.8129x + 103.56$	0.71
Calcite	$y = -0.0002x + 0.0277$	0.83	$y = 0.6627x + 292.11$	0.01

Chemical composition

The results obtained from dielectric permittivity and chemical composition by XRF have been compared with linear regression models and multiple correlation diagrams. Only nine elements were studied from the XRF results, eliminating elements with a low proportion (trace chemical compounds). The chemical compounds studied are SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, K₂O, TiO₂, and MnO. During the analysis, silicate rocks and carbonate rocks were studied separately to know the impact of CaO or MgO on silicate rocks and carbonate rocks, respectively (Table 9). Moderate correlations with $\varepsilon''/\varepsilon'$ have been found with SiO₂, Fe₂O₃, and TiO₂. In the case of SiO₂, a greater presence of this compound results in a reduction in the loss tangent values, by contrast, in the Fe₂O₃ and TiO₂ compounds, an increase in the loss tangent values is observed. In this case, the results found are consistent with previous studies [23].

A similar study was carried out, correlating the mentioned chemical compounds obtained by XRF and the T70 parameter (Table 9). For silicate rocks, high-moderate correlations of elements that promote heating (with a negative slope of the T70 correlation) have been found for Al₂O₃, Fe₂O₃, as well as for CaO, MgO, TiO₂, and MnO. On the other hand, SiO₂ negatively affects heating in silicate rocks, as the slope of the correlation line is positive. For carbonatic rocks, the compounds CaO and MgO have been studied, both showing a high correlation, and it is observed that CaO affects negatively heating (positive slope of the T70 correlation), and MgO affects it positively (negative slope). The results obtained on the CaO are interesting since all studies carried out so far indicated that this element negatively affects heating [28,30,31]. This result is because they only studied carbonate rocks or carbonate minerals. Nevertheless, it should be noted that calcium was never taken into account for silicates, and this element is present in several minerals, such as pyroxene.

Table 9. Equations and Pearson's correlation coefficients for the chemical compounds described by XRF for loss tangent and parameter T70.

Chemical compounds	ϵ''/ϵ'		T70	
	Equation	R ²	Equation	R ²
Fe ₂ O ₃	$y = 0.0011x + 0.0044$	0.31	$y = -21.271x + 291.27$	0.57
SiO ₂	$y = -0.0003x + 0.0295$	0.39	$y = 5.6815x - 195.5$	0.74
Al ₂ O ₃	$y = 0.0007x + 0.002$	0.20	$y = -18.436x + 421.37$	0.71
Na ₂ O	$y = 0.003x + 0.0052$	0.23	$y = -29.676x + 190.98$	0.28
K ₂ O	$y = 0.0007x + 0.0112$	0.02	$y = -15.699x + 168.5$	0.06
TiO ₂	$y = 0.0091x + 0.0046$	0.35	$y = -135.3x + 252.85$	0.39
MnO	$y = 0.1275x + 0.0074$	0.08	$y = -1789.7x + 193.64$	0.35
CaO (silicates)	$y = -0.0001x + 0.0138$	0.14	$y = -8.4195x + 161.61$	0.34
MgO (silicates)	$y = 0.0011x + 0.0084$	0.16	$y = -14.197x + 174.88$	0.35
CaO (carbonates)	$y = 9E-05x + 0.0006$	0.22	$y = 10.962x - 246.46$	0.86
MgO (carbonates)	$y = -0.0001x + 0.0054$	0.23	$y = -13.448x + 363.38$	0.85

Physical properties: the crystal size

A factor of differential heating found in the literature is the size of the crystals that form the rock, giving rise to heterogeneity in the sample and causing deficiencies in heat transmission [26,27]. Therefore, the results obtained from the dielectric permittivity (loss tangent) and the crystal size data obtained by the imaging technique have been compared to analyse the influence of crystal size.

The correlations observed in this study are low, obtaining inconclusive results due to the interference of multiple minerals. It has only been possible to establish correlations in mono-mineral rocks. As there is no interference between the different mineral phases, moderate correlations have been obtained, which indicate a reduction in the tangent values of loss / increase of T70 in calcite and quartz minerals with a larger crystal size (Fig. 8). Nicco [46] studies carried out on granites provided similar results to those found in this work.

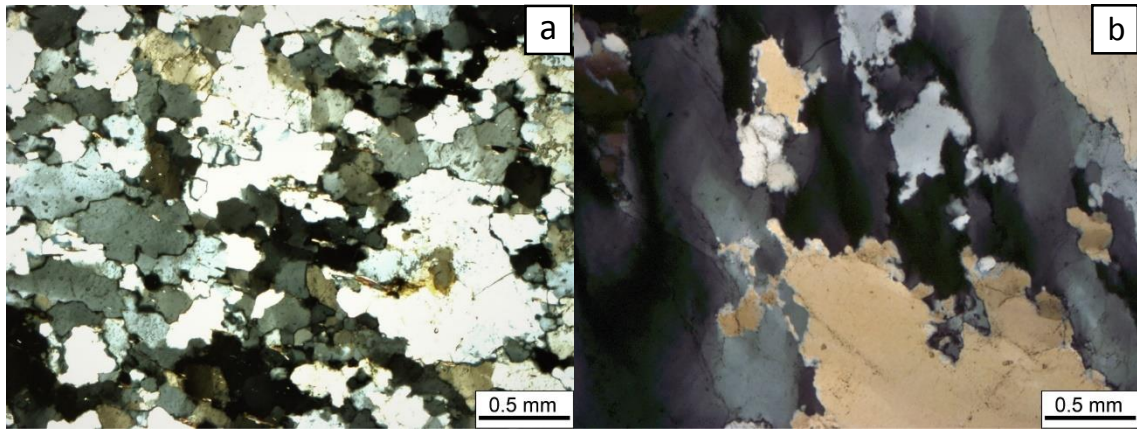


Fig. 8: Images of thin sections (crossed nicols) of **a)** quartzite-1 (QRT-1) and **b)** quartzite-2 (QRT-2).

The samples QRT-1 and QRT-2 are made up of quartz in more than 95%, which is why they are considered quartz mono-mineral rocks. The only discrepancy between them is the crystal size with a mean size for the QRT-1 sample of 0.1 mm^2 and the QRT-2 sample of 0.25 mm^2 . After this, there are discrepancies in the T70 parameter, obtaining values of 289.7 for QRT-1 and 504.2 for QRT-2. It is observed that rocks with a larger crystal size experience longer heating times for the same composition. Something similar can be observed in the samples CAL-1, CAL-2, and CAL-3. All of them are micritic limestones (microcrystals) with different proportions of sparite crystals with a size of 0.05 mm^2 . The CAL-1 and CAL-2 samples have 15-20% sparite and the CAL-3 sample 30%. As in quartzites, discrepancies have been observed in the T70 values for the samples with values of 310.9 for CAL-1, 334.5 for CAL-2, and 426.7 for CAL-3.

Physical properties: apparent density

The data of the apparent density for the 24 samples studied were related to the dielectric permittivity (loss tangent). In this case, a linear regression model has been performed (Fig. 9). The correlation between density and dielectric permittivity (loss tangent) is low, and there is no correlation between these two parameters.

Moreover, the relationship between density and heating of materials with microwaves has been studied. As in the previous case, the results obtained from heating with microwaves (T70) and the apparent density data have been compared, performing a linear regression model (Fig. 10). The correlation between density and heating is low,

and there is no relationship between these parameters, contrary to what has been reported by other authors [38]. This discrepancy may be due to several reasons, including the test method or the samples used. In this study, mainly silicate samples in powder format have been used, while in other studies [38], a mixture of silicate and metallic minerals were tested.

Some studies [33] indicate that density is one parameter that affects the loss tangent; these results can be altered in materials with low porosity. Porosity can promote heating since the pores can contain water in different states [21,40]. In the case of road aggregates, porosity is harmful and normally avoided in road pavements.

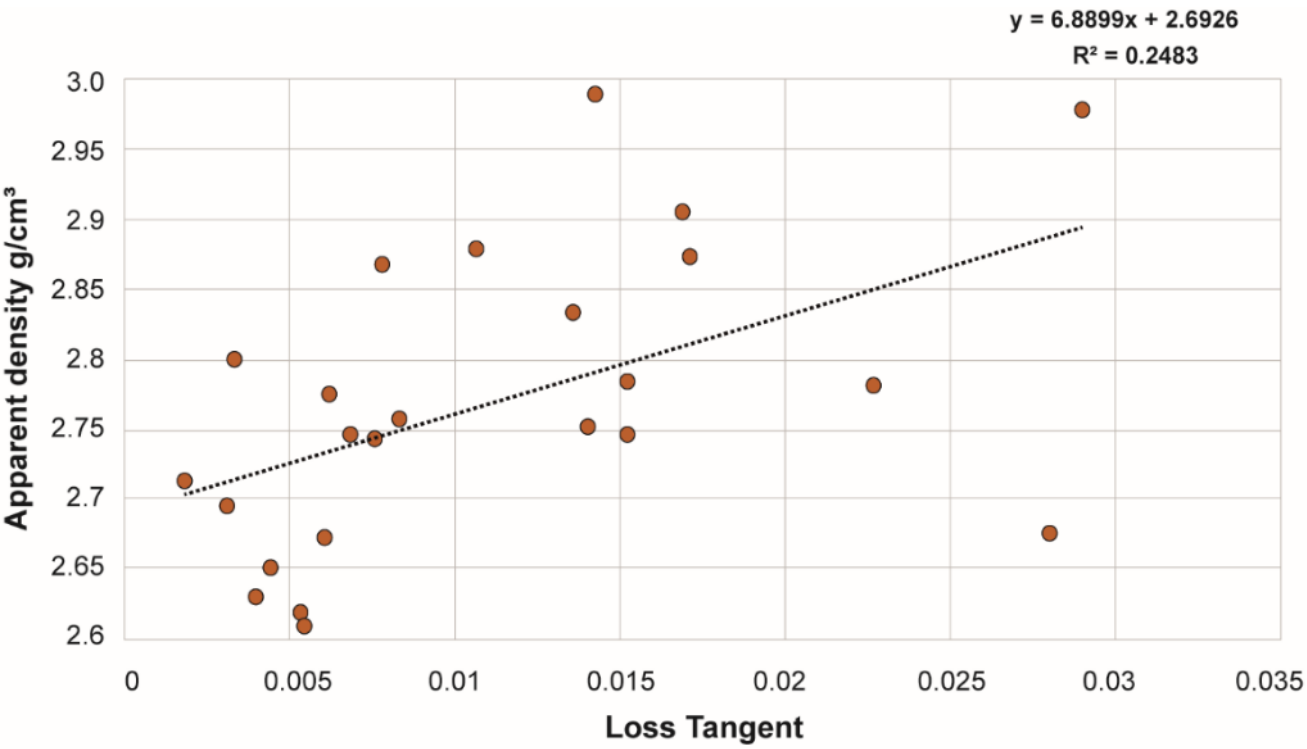


Fig. 9: Dielectric permittivity values versus apparent density.

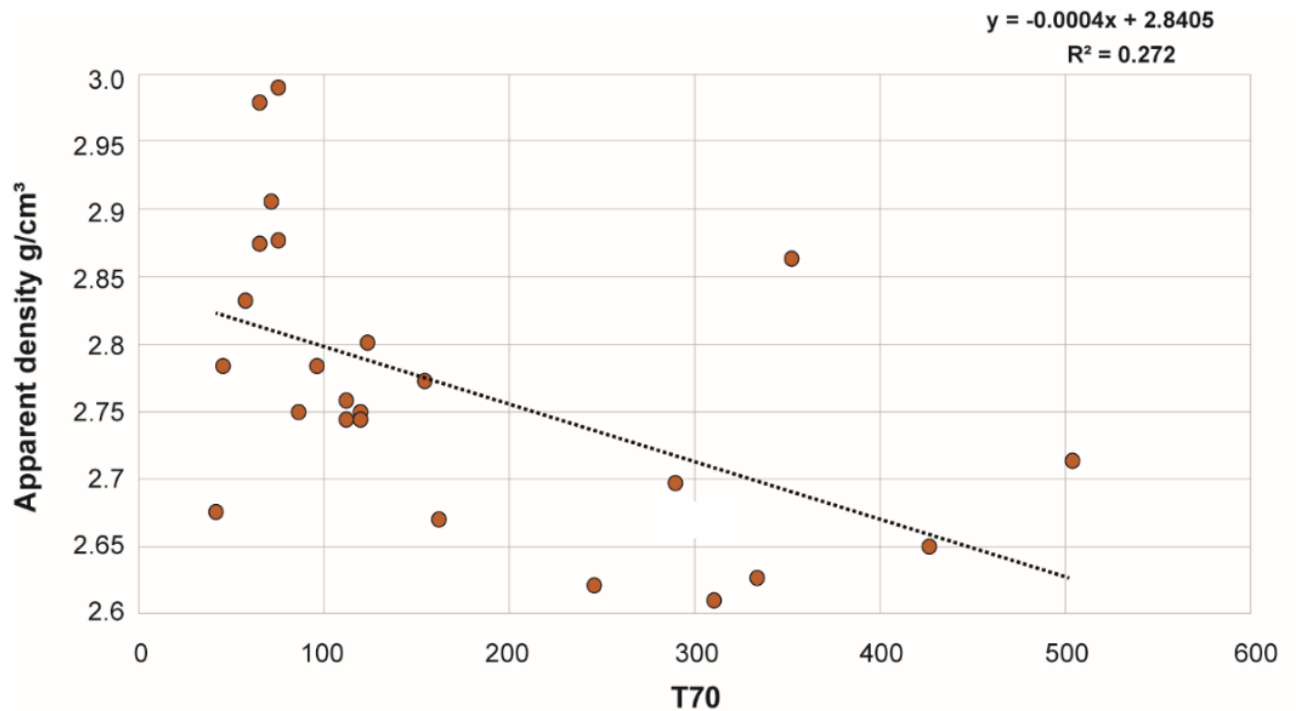


Fig. 10: T70 values versus apparent density.

4.2. A model to predict the microwave susceptibility of the rocks based on their composition

Multiple regression analyses have been performed to apply this knowledge to any rock susceptible to being used as aggregate for asphalt mixtures. The search for the models has been carried out for two microwave susceptibility parameters: parameter T70 and the loss tangent. The chemical and physical properties were analysed as possible causes of differential heating. The most likely causes of differential heating are chemistry and mineralogy. For this reason, models will be made for chemistry and mineralogy, leaving aside the crystal size and the apparent density.

The first two models were made for direct heating data (parameter T70): the first for mineralogical data obtained with the XRD test and the second for chemical elements obtained with the XRF test. With these models, it would be possible to predict the microwave susceptibility that any rock has, including it in one of the categories of parameter T70.

For the study of the influence of mineralogy on heating, eleven variables (minerals) have been used in the model, which are quartz, plagioclase, k-feldspar, biotite, muscovite, chlorite, hornblende, pyroxene, olivine, calcite, and dolomite.

Some of the variables were discarded from the study because they presented autocorrelation with other variables; they were a linear combination of them or were not statistically significant. As a result, only the following minerals were introduced in the model: calcite (Cal), quartz (Qz), chlorite (Chl), pyroxene (Px), and hornblende (Hbl). As a result, the adjusted coefficient of determination (R^2) obtained is 0.7963, and the equation obtained is the following:

$$T70 = 114.376 + 2.58537 \times Cal + 0.575402 \times Qz - 1.22743 \times Chl - 0.952189 \times Px - 1.74457 \times Hbl \quad (2)$$

A similar process has been followed with XRF data to obtain a model for the chemical data; 10 variables (elements) analysed during the phase of the influence of each element have been used in the model, which are SiO_2 , Al_2O_3 , Fe_2O_3 , CaO, MgO, Na_2O , K_2O , TiO_2 , and MnO.

Some of the variables were removed from the model due to a high autocorrelation with other variables due to a linear combination of these or because they had no statistically significant relevance. Nevertheless, the following elements are part of the model: carbonate (CaO), silica (SiO_2), aluminium (Al_2O_3), and iron (Fe_2O_3). The adjusted coefficient of determination (R^2) obtained is 0.8633, and the equation obtained is the following:

$$T70 = 300.827 + 1.11453 \times CaO + 0.22899 \times SiO_2 - 7.52744 \times Al_2O_3 - 10.6784 \times Fe_2O_3 \quad (3)$$

Next, the same process was carried out for the loss tangent. The analyses were performed for the mineralogical data obtained with an XRD test and chemical elements obtained with an XRF test. For the study of mineralogy, the 11 same variables have been used to calculate the model for the parameter T70. Part of the variables was discarded

from the model since they were not statistically significant or presented autocorrelation with other variables. Therefore, they have been used: plagioclase (Plg), muscovite (Ms), chlorite (Chl), and pyroxene (Px). The adjusted coefficient of determination (R^2) obtained is 0.5135, and the equation obtained is the following:

$$\begin{aligned} Loss\ Tangent = & 0.00484136 + 0.000191515 \times Plg - 0.000164382 \times Ms + \\ & 0.000443923 \times Chl - 0.00023827 \times Px \end{aligned} \quad (4)$$

The same process has been followed to study the variables chemical elements. Some variables were eliminated from the model due to the lack of statistical significance or high autocorrelation with other variables. Nevertheless, the following elements are part of the model: silica (SiO_2), aluminium (Al_2O_3), iron (Fe_2O_3), sodium (Na_2O), and potassium (K_2O). The adjusted coefficient of determination (R^2) obtained is 0.6544, and the equation obtained is the following:

$$\begin{aligned} Loss\ Tangent = & 0.00415403 - 0.00000152257 \times SiO_2 - 0.00214932 \times Al_2O_3 \\ & + 0.00247492 \times Fe_2O_3 + 0.00553521 \times Na_2O + 0.00425194 \times K_2O \end{aligned} \quad (5)$$

The best correlations between the different physical and chemical properties have been obtained with the direct heating method (parameter T70). The models elaborated with the permittivity values could also be used since moderate correlation coefficients are obtained. In addition, other studies have shown a high correlation between the T70 parameter and the loss tangent values [19].

4.3. Estimation of the influence of physical and chemical properties of the rocks on microwave heating

The models made with the T70 parameter and the loss tangent allow us to observe that higher correlations are obtained with the T70 parameter (Table 10). This result is advantageous since this measurement technique is easy to apply and is a rapid and low-cost technology. With the models carried out for chemistry and mineralogy with the T70 parameter, 86.33% and 79.63% of the variability present in the T70 can be explained. However, other variables such as crystal size, thermal conductivity [57,58],

and electrical conductivity [59,60] of the aggregates can affect heating. From the coefficients of determination obtained, it is observed that the factor that most influences microwave heating is the chemistry of the materials, followed by mineralogy (closely associated with the chemistry of the rocks).

Table 10. Comparison of parameter T70 versus loss tangent.

	T70	ϵ''/ϵ'
Coefficient of determination for mineralogy	0.7963	0.5135
Coefficient of determination for chemistry	0.8633	0.6544
Necessary equipment	Microwave	Dielectric properties equipment
Sample preparation	Aggregate 4/8	Grinding
Results analysis	Direct	Require interpretation
Cost	Low	Expensive

Chemical elements such as Al_2O_3 and Fe_2O_3 positively affect the heating of aggregates for road construction (Table 9 and Equation 3). Other elements, i.e., MgO , TiO_2 , and MnO , seem to promote heating (despite their moderate-low R^2 values in Table 9). Nevertheless, their low content in stable aggregates for pavement construction and maintenance recommended not to consider them in the elaboration of Equation 3. On the other hand, chemical elements such as SiO_2 and CaO (in carbonate rocks) slow down the heating (Table 9 and Equation 3). Other authors have reported that part of these considerations on chemistry, chemical elements such as Fe_2O_3 [21,23,28,31] were considered one of the great promoters of microwave heating. However, this influence is low in silicate minerals, and Fe_2O_3 improves the heating performance when it is part of metallic minerals, impurities [12], or residues such as slag [30]. Other chemical elements such as MnO or TiO_2 were also presumed to be high microwave absorbers [21,28,32], but their influence on aggregates is low since they have little representativeness. Therefore, the literature considered other elements such as Al_2O_3 and MgO as medium microwave absorbers [25,29]. However, both resulted in high microwave absorbers in silicate rocks in this study, reducing the heating time. Concerning CaO , it has always been considered a low microwave absorber [28,30,31] because it was only studied in carbonate minerals. However, when it is associated with Al_2O_3 , MgO , and Fe_2O_3 in silicate minerals, it does not slow down the heating, so

together with these compounds, it is a harmless element for microwave applications. Finally, the elements of SiO₂ and CaO (in carbonate minerals) are considered low absorption, described in the literature by other authors [28,30,31]. The rest of the elements, such as CaO (in silicate minerals), K₂O, and Na₂O, do not affect mineral heating, although previously, Na₂O was considered a medium absorber [28,32].

Minerals that promote heating with microwaves are pyroxene, chlorite, biotite, and hornblende, these contain significant concentrations of heating-promoting chemicals such as Al₂O₃, Fe₂O₃, and MgO, and some of them contain water in their mineral structure [21] (Table 11). Some of these minerals, such as biotite and pyroxene, had been reported as minerals that favour mineral heating with microwaves [12,16,21], but the causes of this rapid heating had not been reported. In contrast, minerals such as quartz, calcite, and muscovite are considered weak microwave absorbers and slow down heating [12,16,21]. Other minerals such as plagioclase or feldspar are considered inert at the rate of heating.

Table 11. Chemical composition of minerals with high microwave response.

Mineral	Mineral formula
Pyroxene	(Ca,Mg,Fe) ₂ (Si,Al) ₂ O ₆
Chlorite	(Mg,Fe) ₃ (Si,Al) ₄ O ₁₀ (OH) ₂ ·(Mg,Fe) ₃ (OH) ₆
Biotite	K(Mg,Fe) ₃ AlSi ₃ O ₁₀ (OH, F) ₂
Hornblende	Ca ₂ (Mg,Fe,Al) ₅ (Al,Si) ₈ O ₂₂ (OH) ₂

Regarding the apparent density of the material and microwave heating, no relationship has been found. This may be due to the lack of correlation between both parameters or to discrepancies between other studies due to the use of different test methods or the type of samples used.

About the size of the crystals, moderate correlations have been obtained for quartz and calcite. The increase in the size of these minerals (minerals inert or with low susceptibility to microwaves) would produce slower heating. An increase in size can cause the microwave waves to be distributed more inefficiently and, therefore, the heating to be slower. This study has also demonstrated that two quartzite rocks (QRT-1 and QRT-2) with the same composition but different crystal sizes, QRT-1 fine grain and QRT-2 coarse grain, present different heating rates (higher for the QRT-2). In both cases, the heating rate is slow since they only consist of quartz, a mineral that has already been

considered inefficient but presents a slower heating rate when presenting a larger size [24].

It is possible that minerals that present rapid heating, such as pyroxene or chlorite, have faster heating when they present large crystal sizes (for example, poikilotopic textures). This can be intuited in the OPH-1 and OPH-4 samples, although it has not been statistically demonstrated due to the influence of other minerals. In the case of minerals with high heating rates and large crystals (poikilotopic texture), the distribution of microwave waves is homogeneous, and this is the main factor to improve the homogeneous heating in the rock [35,36].

These results will have a significant impact on the search for aggregates that present high microwave heating for self-healing applications [6,7], heating of aggregates for industry [41], and mining [27]. It will also save economic costs derived from maintenance or energy expenses [13] and environmental costs caused by CO₂ due to the burning of fossil fuels [5,13].

5. Conclusions

This research investigates the factors that affect the differential heating or susceptibility to microwaves of aggregates for road pavements. Different laboratory tests have studied the chemical properties (mineralogy and chemical) and physical properties (crystal size and apparent density) of the aggregates.

The dielectric permittivity (loss tangent) and the direct heating values (parameter T70) of the 24 aggregates studied have been correlated with each of the properties analysed. Predictive models of heating from elemental composition and mineralogy have been developed using linear and multiple correlation diagrams.

After analysing the results of this study, it is proved that the type and origin of the materials strongly affect the microwave heating process, obtaining the following conclusions:

- Mineral of high susceptibility favouring heating is especially pyroxene. Chlorite, biotite, and hornblende probably promote microwave heating, although additional research is needed to confirm this influence. On the other hand, minerals with low microwave susceptibility are quartz and muscovite. Since calcite is present at high contents in carbonatic rocks, it is not a good predictor of microwave susceptibility in this type of aggregates.
- The content of Al_2O_3 and Fe_2O_3 affects heating positively. The elements MgO , TiO_2 , and MnO , seem to promote heating, but their content in pavement aggregates are not representative. Elements such as SiO_2 and CaO (in carbonate rocks) negatively affect heating. Other chemical elements such as Na_2O , K_2O , and CaO (in silicate minerals) can be considered inert in microwave heating.
- Regarding the physical properties, the study of the size of the crystals was inconclusive due to the interference of multiple minerals. Nevertheless, in the case of mono-mineral rocks with low heating rate minerals, as the size of the crystals increases, the microwave heating gets slower.
- No relationship was found between the density data of the aggregates under study and their microwave heating data, possibly due to the low

porosity of the aggregates for pavements, the absence of water in the pores, or a discrepancy between other studies in the test method or the samples used.

As a result, the chemical and mineralogical properties of the material are closely related to differential microwave heating. Multiple regression models have been elaborated to deduce these relationships, obtaining a strong correlation for the chemistry and mineralogy of the rock versus the T70 parameter. However, the regression models versus the dielectric loss tangent were weaker. Therefore, it is recommended to apply the T70 prediction models to estimate the microwave susceptibility of aggregates.

The novelty of this paper is the study of the heating of a large number of aggregates samples corresponding to different lithologies. In addition, knowledge has been obtained on the natural factors (chemical and physical) that affect the heating of aggregates and cause the differential heating of the different lithologies. This new information helps dry aggregates facilities, on site de-icing of roads, self-repair techniques of asphalt mixtures, and other applications to aggregates or pavements that can be implemented with microwaves.

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