Self-healing of asphalt mixture: the impact of the minerals forming the aggregates in the efficiency of the heating by microwaves.

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Abstract. The current concern for the environment and the need to reduce greenhouse gas emissions have led to new technologies related to microwave energy. One of these technologies is the self-healing of asphalt mixtures, which consists of repairing pavements through microwave application on the surface, avoiding premature road failure. Asphalt mixtures for roads are made up of more than 90% by weight of aggregates of different compositions and origins, in addition to a bituminous binder and sometimes additives. From other studies, it is known that the physical behaviour of aggregates is a function of their composition, that is, of their minerals and their proportions. Microwave heating of aggregates has proven to be an effective technique, but there are gaps in understanding how microwaves interact with aggregates and the reasons for their differential heating.

This research has studied 18 minerals that are commonly part of the rocks used as road aggregates. The objective is to identify the minerals that present the best heating rates to relate them to the differential heating of aggregates for roads. The results obtained are promising, facilitating the understanding of microwave heating of minerals. Regarding chemical composition, elements such as MgO, MnO, TiO, Al₂O₃, Fe₂O₃, and CaO (in silicate minerals) favour the heating of minerals and other elements such as SiO₂ and K₂O Na₂O, and CaO (in carbonate minerals) retard the heating. Regarding the physical properties, density and habit of the minerals do not influence the heating, but other properties, such as the diaphaneity and the size of the crystals, influence the heating with microwaves.

1. Introduction

Preventive maintenance of road structures is under development to extend the useful life of pavements, mainly due to environmental and economic benefits [1,2]. The process known as self-healing is a natural process that restores the initial properties of the pavement by healing microcracks produced by traffic and weather conditions [3]. Self-healing occurs naturally, but it requires extended periods of rest and temperatures close to 70 °C [4]; therefore, assisted heating is required (for example, with microwave) [5]. Furthermore, microwave energy is widely used in industrial applications, such as rock crushing, drying, and mineral separation [6].
Electromagnetic waves cause the heating of matter due to the friction of the molecules, causing heating from the inside of the material (volumetric heating), the characteristics of the material must also be taken into account according to its affinity with microwaves: a) insulating or transparent, without heating, b) conductive or metallic, only heated externally and c) absorbent or dielectric, suffer volumetric heating [6].

Bituminous pavements are made up of a mixture of aggregates with specific characteristics, which represent more than 90% by weight of the mixture, a bituminous binder and sometimes additives that improve some of the properties of the pavement. The results obtained by other authors show that the bituminous binder is transparent to microwaves [7]. From the point of view of aggregates, there are recent studies related to heating [8] or Li et al. [9], some of them dealing with self-healing in pavements [5]. These studies classify the rocks according to their response to microwave radiation, studying the different origin and composition of the aggregates. It is observed that the composition of the rocks plays a key role in their differential heating. These studies on aggregates are fundamental and allow us to know the interaction of microwaves with rocky materials. However, the susceptibility of rocks is a function of the behaviour of the minerals that make up the rock (composition and proportion [6]. The heating of each mineral must be considered individually to gain a greater understanding of how microwave energy interacts with rocks. The interaction of the microwave with minerals is little studied. Most of them were carried out on metallic minerals, and silicate minerals were not incorporated in the studies since they were considered transparent to microwaves, so it is necessary to study the rock-forming minerals (silicate minerals) in detail. The studies found were mainly focused on testing the heating rate of the minerals, measuring the temperature at different intervals, to finally categorize each mineral according to its susceptibility (high, medium, or low). These tests are performed at different microwave frequencies and powers, resulting in heterogeneous results.

The first study carried out was that of Ford and Pei [10], where 17 oxides were studied using a microwave with a frequency of 2.45 GHz and a maximum power of 1.6 kW. The results suggest that the dark-coloured compounds heated rapidly, while the light-coloured compounds heated slower but reached higher temperatures. Chen et al. [11] studied 40 minerals in a microwave with a power of 800 watts with heating cycles of 5 minutes. They observed that most of the silicates, carbonates, and sulfates (rock-forming minerals) are transparent to microwave energy, but other (metal) oxides heat up quickly. Walkiewicz et al. [12] investigated silicate minerals and metal oxides at 2.45 GHz, which resulted in slow heating rates for silicate minerals and high heating rates for metallic minerals. A similar result was obtained by Tinga [13] on metallic oxides, classifying them according to their susceptibility as hyperactive, active, difficult to heat and inactive. The work of Harrison [14] was one of the first to study several silicate minerals, including 25 minerals and a microwave with a power of 650 W for 180 seconds, and made a classification according to their heating rate. Lu et al. [15] used a 2 kW multimode cavity to heat eleven basic rock-forming minerals for 3 minutes, relating heating to the iron content. Finally, Zheng et al. [16] studied the heating of silicate minerals and related them to some chemical and electrical properties. At the same time, the mining industry has spent years studying the thermal properties of different minerals to optimize mineral processing [17].

This work aims to identify the minerals with the best heating rates and to serve as knowledge to know how it affects the heating of aggregates for industry and construction. For this reason, a set of common minerals has been chosen that enter the composition of most carbonate and silicate rocks. The optimal heating temperature for each mineral will be determined using the direct heating technique, known as the T70 parameter (which is the time it takes for a sample to reach a temperature of 70 °C) [5]. Subsequently, the direct heating data will be compared with the physical properties (density, diaphaneity, and habit) and chemical properties to know which parameters influence the heating.
2. Materials and Methods

The samples correspond to 18 minerals Figure 1, which are commonly part of the mineralogical composition of the road aggregates. They have been classified into two groups according to their chemical composition (Table 1). In this way, it is possible to compare microwave heating efficiency based on their different physical properties (habit, density, and diaphaneity) or chemical compositions.

To study the heating produced by microwave energy in the different minerals, the authors followed a test protocol described in [5] that measured the heating efficiency of each aggregate; when using pure minerals without porosity, it was not necessary to dry them in an oven, it was verified that they did not have water on the surface. In this process, a 20L Orbeegozo MI-2014 microwave oven was used, with an output power of 700W and a frequency of 2.45 GHz (Figure 2). In addition, a silicon mat replaced the interior plate. The test measured the temperature with an infrared temperature gun (Testo 830-T1), heating the material at fixed time intervals (every 10 s) until reaching a surface temperature higher than 110 °C or 60s. In the design of the experiment, the principle of randomization was followed, and the authors repeated the experiment 30 times to avoid variability underestimation. Subsequently, the diagnosis of the data was verified to ensure that they were correct (homoscedasticity, normality, and independence).

![Figure 1. Minerals under study.](image-url)
Table 1. Minerals in the study have chemical and physical characteristics: density (g/cm³), diaphaneity, and habit.

| Mineral                     | Formula          | Density | Diaphaneity | Habit      |
|----------------------------|------------------|---------|-------------|------------|
| Quartz (macrocrystalline)  | SiO₂             | 2.65    | Transparent | Prismatic  |
| Quartz (microcrystalline)  | SiO₂             | 2.65    | Transparent | Prismatic  |
| Silex (cryptocrystalline)  | SiO₂             | 2.65    | Translucent | Massive    |
| Orthoclase                 | KAlSi₃O₈         | 2.56    | Translucent | Prismatic  |
| Albite                     | NaAlSi₃O₈        | 2.63    | Translucent | Prismatic  |
| Biotite                    | K(Mg,Fe)₃AlSi₃O₁₀(OH,F)₂ | 3       | Translucent | Lamellar   |
| Muscovite                  | KAl₂(AlSi₃O₁₀)(OH)₂ | 2.8     | Transparent | Lamellar   |
| Chlorite                   | (Mg,Fe²⁺)₃Al(Si₃Al)O₁₀(OH)₈ | 2.65    | Translucent | Lamellar   |
| Actinolite                 | Ca₂(Mg,Fe²⁺)₂Si₈O₁₈(OH)₂ | 3       | Opaque      | Fibrous    |
| Hornblende                 | Ca₂(Mg,Fe,Al)₃(Al,Si₃)O₇(OH)₂ | 3.2     | Translucent | Prismatic  |
| Olivine                    | (Mg,Fe)₂SiO₄     | 4.4     | Translucent | Prismatic  |
| Garnet                     | Fe²⁺₃Al₃(SiO₃)₃ | 4.2     | Translucent | Granular   |
| Tourmaline                 | Na₂Mg₃Al₆[(BO₃)₃(Si₆O₁₈)].(OH)₄ | 3.15    | Opaque      | Columnar   |
| Calcite                    | CaCO₃            | 2.71    | Transparent | Prismatic  |
| Aragonite                  | CaCO₃            | 2.93    | Translucent | Columnar   |
| Dolomite                   | CaMg(CO₃)₂       | 2.85    | Opaque      | Massive    |
| Magnesite                  | MgCO₃            | 2.71    | Opaque      | Massive    |
| Rutile                     | TiO₂             | 4.25    | Opaque      | Prismatic  |

Figure 2. Equipment used to perform temperature measurements.

3. Results and Discussion
The heating results of the samples under microwave radiation are shown in Figure 3. Based on this data, the slope of the estimated regression line representing the susceptibility of the mineral to be heated by microwave radiation was extracted. In this way, the parameter T70 can be calculated. It is the time in seconds that the sample requires to reach 70 °C from an ambient temperature of 20 °C by microwave radiation, with an output power of 700W and a frequency of 2.45 GHz; this parameter has already proven its worth in other [5,18] T70 allows materials to be classified into five classes; the first...
class corresponds to samples with very rapid heating: 70 °C in less than 25 s. The second class includes samples that require between 25-50 s to reach 70 °C. Samples of the third class, with medium heating, reaching 70 °C between 50-100 s. Samples of the fourth class (slow heating) need between 100 and 200 s to reach 70 °C. Finally, the fifth class includes samples that require more than 200 s to reach 70 °C with prolonged heating.

The minerals under study have been classified according to their T70, and the result can be seen in Figure 4, indicating the T70 value obtained for each mineral.

- Samples with high susceptibility (classes 1 and 2): rutile (7.1), magnesite (7.5), dolomite (10.5), biotite (17.6), hornblende (18.7), garnet (22.4), and chlorite (34.3).
- Samples with medium susceptibility (class 3): actinolite (53.1), muscovite (73.3), olivine (74.8), and calcite (75.2).
- Samples with low susceptibility (classes 4 and 5): aragonite (130.2), quartz cryptocrystalline (197.5), quartz microcrystalline (230.7), tourmaline (238.4), quartz macrocrystalline (416.0), orthoclase (531.9), and albite (807.8).

Figure 3. Heating graph of the minerals under study.

Figure 4. Classification of minerals according to parameter T70 (not to scale).
A detailed study of the physical and chemical properties of the minerals understudy has been carried out to define the causes of differential heating. First, concerning the physical properties, the relationship between habit and the T70 parameter was studied. In this case, no relationship is found except for the crystal size and heating. It is observed in quartz minerals (macrocrystalline, microcrystalline and cryptocrystalline) or between calcite-aragonite. It is observed that for the same chemistry SiO2 for quartz and CaCO3 for calcite-aragonite, the heating times are shorter at a smaller crystal size.

For the relationship between diaphaneity (transparent, translucent, and opaque) and T70, a Mann-Whitney U test was performed, obtaining significant differences between transparent-opaque and translucent-opaque minerals. For this reason, diaphaneity affects the heating, obtaining shorter heating times for opaque minerals. No significant differences have been found for transparent-translucent minerals, although heating times are shorter for translucent minerals. Other authors reported similar findings when they studied the influence of mineral colour [14].

Regarding the relationship between density and the T70 parameter, linear correlation studies have been carried out using Pearson's correlation coefficient. A correlation R² = 0.15 has been obtained, so there is no correlation between both variables, refuting the results provided by other authors such as [19]. The disparity between the results of [19] and this work can be explained by using different minerals, such as oxides or because the minerals used had porosity [20].

Finally, multiple correlation diagrams have been made between chemistry and the T70 parameter. The results show that some chemical elements promote heating, and others slow it down. Among the elements that promote heating in minerals, we find elements such as MgO, MnO and TiO. As elements that slightly promote heating, we see Al2O3, Fe2O3, and CaO (silicate minerals). Instead, chemical components such as SiO2, Na2O, K2O, and CaO (carbonate minerals) slow down the heating.

4. Conclusions

Regarding the physical properties studied and their relationship with heating, the habit of minerals does not influence heating. Still, the crystal size affects minerals such as quartz (macrocrystalline, microcrystalline and cryptocrystalline) or calcite and aragonite. The smaller the crystal size, the higher the heating rates.

The diaphaneity of the mineral influences the heating of the mineral, obtaining shorter heating times for opaque minerals followed by translucent and transparent ones. Regarding density, there is no relationship between greater heating and a greater or lesser density of the minerals.

With chemistry, there are chemical compounds that slow down the heating, like SiO2, K2O, Na2O, and CaO (in carbonate minerals), but there are also elements that speed up the heating, like MgO, MnO, and TiO. Other chemicals such as Al2O3, Fe2O3, and CaO (in silicate minerals) also slightly accelerate heating. Impurities in some minerals and their volume may have caused higher temperatures, as in calcite.

Minerals have been classified into three categories based on their absorption/susceptibility to microwaves.

- Absorbing minerals: biotite, chlorite, hornblende, garnet, dolomite, magnesite, and rutile. They reach a high temperature in a short period of time.
- Medium absorbing minerals: muscovite, actinolite, olivine and calcite. They heat up but require more time than the previous group.
- Non-absorbent minerals: quartz (macrocrystalline, microcrystalline, and cryptocrystalline), orthoclase, albite, tourmaline, and aragonite. The speed of heating is very slow.

The study of mineral heating is challenging and requires many additional studies, as chemical variations, and mineral habit can alter heating results. Knowing how microwave heating works in minerals will serve to know in detail how heating works in aggregates for industrial and technological applications such as rock drying, mineral grinding or self-healing.
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