MICROWAVES AS AN ENERGY SOURCE FOR PRODUCING ZEOLITES
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ABSTRACT
"Sol-Gel" is one method for producing synthetically zeolites from sodium hydroxide, sodium hydroxialuminate and sodium silicate in an aqueous solution at temperatures around 85°C. Some zeolites are resistant to temperatures up to 800°C, therefore the proposal consisted in heating up mixtures of the same dry compounds with microwaves (2.45 GHz). This work presents a summary of the results where tests of 20 minutes at temperatures between 700°C and 750°C produced Faujasite, which is a zeolite.

INTRODUCTION
Certain minerals seem like they were boiling when they are warmed up to certain temperatures. These minerals are known as "zeolites" from the Greek "zeo" to boil and "lithos" stone. The zeolites are hydrated crystalline aluminosilicates, that conform a porous structure with diameter of pores between 3-10 Å. The structure of each unitary cell of a zeolite is constituted of tetrahedrons forming blocks that can be united with others. The atoms of oxygen form the four vertices of each tetrahedron, therefore each atom of silicon or four atoms of oxygen can surround aluminum. Hence, the units of primary construction of the zeolites are the tetrahedrons (SiO₄) and (AlO₄) sharing vertices and forming oxygen bridges. The tetrahedrons could be united sharing the two, three or four vertices, forming a great variety of different structures. Many zeolites are based on a unit of secondary construction that consists of 24 tetrahedrons of united silicon or aluminum. Their final shape is like a honeycomb framework with interconnected channels, Figure 1. This material is highly crystalline and the array of cavities can be occupied by ions, such as Ca, Na and K, and also water. The structure of the zeolites allows them to interchange ions, reason why, among many other applications, they are used as catalyst. Veins and cavities of basic igneous rocks, especially basalt are one of the most common sources of natural zeolites, although for having more uniform properties it is also common to produce them synthetically. One of the employed methods is known as "Sol-Gel", which consists of starting from three aqueous solutions: sodium hydroxide (NaOH), sodium hydroxialuminate (NaAl(OH)₄) and sodium silicate (Na₂SiO₃), which are typical compounds. These compounds are poured until the solution becomes cloudy, with a part gelatinous and viscous, named "Gel".

The proposal of this investigation was to use microwaves as an energy source for producing zeolites from the raw materials that typically would be used in the "Sol-Gel" process. It is considered in this work that some zeolites can resist temperatures up to 800°C without loosing their structure and knowing that microwaves can be used as an energy source for producing certain ceramics, the objective of this work is to use a mixture of materials similar to that used in the "Sol-Gel" method for producing zeolites with microwaves at 2.45 GHz.
Figure 1. Structure of a zeolite, known as “Faujasite”, which was found in this work. Either, aluminum or silicon atoms are located over the vertices and oxygen is in between, as shown.

The reagents (in dust) that were used for producing the zeolitic structure were:

Sodium silicate (Na$_2$SiO$_3$*H$_2$O): SiO$_2$ (62 wt%), Na$_2$O (19.6 wt%), H$_2$O (18.5 wt%).
Aluminum hydroxide (Al(OH)$_3$): 95% Purity
Sodium hydroxide (NaOH): 98.25% Purity

Prior to mixture preparation, the raw materials were dried and weighted. The composition was: NaOH: 14.7 wt.%, Al(OH)$_3$: 28.8wt.%, Na$_2$SiO$_3$*H$_2$O: 56.4 wt.%.
7 gr of powders thoroughly mixed in this proportion were placed in an alumina crucible (50 mm diameter, 36 mm internal diameter and 35 mm height). Alumina is relatively transparent to microwaves at temperatures below 600°C.

EXPERIMENTAL
This characteristic is usefully because at least initially, most of the energy is actually going into the mixtures. Experiments were carried out in a microwave kitchen oven (2.45 GHz, 800 Watts; Cavity size: 350mm x 350mm x 240mm), a mapping of this cavity was conducted in order to know where was the best location for placing the crucible. The oven was also modified with the purpose of being able to support long tests and high temperature, which was measured by optical pyrometry. Processing time was 5, 10, 15, 20, 30 and 60 minutes. Samples obtained from the mixtures were characterized by means of X-Ray diffraction in order to identify the resultant crystalline.

RESULTS AND DISCUSSION
Heating by means of microwaves depends on many factors, one of them is the position of the crucible in the cavity, which is related to the electric field that the mixture is exposed, and the ability of the materials for absorbing energy. Preliminary tests consisted in confirming that the materials absorbed microwaves and had some kind of reaction. Weight loss was also determined for estimating stoichiometry from the water loss. Temperature was also monitored.
Tests of 5 minutes showed foam and most of the material was unreacted, maximum temperature in this case was 630°C. Test of 10 minutes showed more foam (Figure 2) and some solid material. Steam was seen in these tests, meaning that foam formation is related to dehydration. Tests of 15 minutes achieved 710°C and exhibited a solid porous material. Temperature evolution is shown in Figure 3 for a test conducted for 60 minutes, but thermal behavior was about the same for all the mixtures, therefore, the achieved temperature for each test can be taken from this graph. After 20 minutes most of the raw material reacted, this was the most promising result, because the tests of 30 to 60 minutes clearly showed a vitreous material that was molten during
the process. Any zeolite that could be formed was destroyed after glass was produced. Figure 4 show the obtained sample after 30 minutes, while no sample was recovered from the tests conducted for 60 minutes, because the molten glass left the crucible by leaking trough its pores. This is also part of the explanation for reducing the temperature after achieving the maximum, part of the absorbing material was not anymore part of the system.

Figure 2. Sample obtained after 10 minutes of exposition. Notice the foam like product that was obtained.

Figure 3. Curve showing thermal evolution of a test conducted for 60 minutes. Measured temperature started at 600°C because that is the minimum temperature that can be read with the optical pyrometer.

Figure 4. Sample obtained after 30 minutes exposition. Maximum temperature was 900°C, but for a very short time.

X-Ray diffraction patterns of the mixtures exposed to microwaves from 5 to 15 minutes showed presence of sodium silicate (Na$_2$SiO$_3$). The only reaction was drying, explaining the formation of foam. Samples obtained after 20 minutes showed a zeolitic phase known as “Faujasite” (Na$_2$Al$_2$Si$_4$O$_{12}$*8H$_2$O) although in a small proportion compared to aluminum hydroxide (Al(OH)$_3$), Figure 5.

Mixtures exposed for 30 to 60 minutes showed glass that was very tide to the crucible an actually molten glass flow through the pores of the crucible. It was not possible to take enough material for conducting reliable X-Ray diffraction characterization, but zeolites are destroyed rather than melted. On the other hand, mixture composition is suitable for producing a low melting point glass.
Figure 5. X-Ray diffraction pattern obtained from a 20 minutes sample, peaks marked with straight lines correspond to Faujasite.

CONCLUSIONS
Tests of 20 minutes produced Faujasite, which is a zeolite, while tests of more than 30 minutes produced glass. It was also found in this work that temperatures under 700°C or above 800°C did not produced any zeolite, thus the best conditions for producing Faujasite are 20 minutes at temperatures between 700°C and 750°C.

REFERENCES
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ACKNOWLEDGEMENTS
Authors express their gratitude to CONACYT (Mexican Council for Science and Technology) and to PAICYT (Program for Science and Technology) of The University of Nuevo León for their financial support. Thanks are also given to Nora Elizondo, at the Physics and Mathematics School at the same university.