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Artículo científico

Study of the synthesis of calcium titanate ($CaTiO_3$) from a gray limestone mineral and titanium dioxide by calcination

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Resumen

Samples of a gray limestone mineral from Coahuila, Mexico were ground, sieved, and washed with a 70 and 100% acetic acid solution. The washed and unwashed mineral was then characterized by X-ray diffraction, X-ray fluorescence and the predominant phase was found to be calcite. A thermodynamic study was performed to evaluate the feasibility of the reaction. Then the samples of this calcite mineral were reacted with reagent-grade titanium oxide in different proportions at temperatures of 600, 700, and 800 °C, for a time of 7 hours. The thermodynamic study shows that this reaction is possible at 400 °C. Washing the mineral with acetic acid does not significantly affect the chemical composition of the mineral. X-ray diffraction analysis indicates the presence of titanium oxide, calcite, and calcium titanate. The best results were found at a temperature of 600 °C, with a molar ratio of two calcium carbonate and one titanium oxide.

Keywords: Mineral; Limestone; Calcium titanate; Thermodynamic study

Resumen

Estudio de la síntesis de titanato de calcio ($CaTiO_3$) a partir de un mineral de caliza gris y dióxido de titanio mediante calcinación. Muestras de un mineral de caliza gris de Coahuila, México, fueron molidas, tamizadas y lavadas con una solución de ácido acético al 70 y 100%. El mineral lavado y sin lavar fue caracterizado por difracción de rayos X, fluorescencia de rayos X y se encontró que la fase predominante era calcita. Se realizó un estudio termodinámico para evaluar la viabilidad de la reacción. Posteriormente, las muestras de este mineral de calcita se hicieron reaccionar con óxido de titanio de grado reactivo en diferentes proporciones a temperaturas de 600, 700 y 800 °C, durante 7 horas. El estudio termodinámico muestra que esta reacción es posible a 400 °C. El lavado del mineral con ácido acético no afecta significativamente su composición química. El análisis de difracción de rayos X indica la presencia de óxido de titanio, calcita y titanato de calcio. Los mejores resultados se encontraron a una temperatura de 600 °C, con una relación molar de dos de carbonato de calcio y un óxido de titanio.

Palabras claves: Mineral; Caliza; Titanato de calcio; Estudio termodinámico

Introduction

Perovskite materials have aroused great interest due to their flexibility to accommodate elements of different ionic radii, allowing them to be stable[1,2]. The growing interest in studying calcium titanate has focused in recent years on taking advantage of its catalytic properties. The development of this process has been strengthened thanks to the current need to generate energy from clean and renewable sources[3,4]. There are various methods to obtain solid inorganic materials, especially oxides. The calcination method is widely used to produce inorganic compounds. It consists of mixing precursor compounds most homogeneously, calculated in stoichiometric quantities, and subjecting said mixture to treatment at high temperatures. This would cause a chemical reaction that generates the product of interest.

Limestone is a sedimentary rock of geological origin with the distinctive formulas of $CaCO_3$ which is known as calcite or calcium carbonate and has a molecular weight of 100.09 g/mol. Its compressive strength varies from 10 to 200 MN/m^2. The hardness of limestone is generally in the range of 2 to 4 Mohs. Physically, it has colors that reflect the levels of impurities present. White deposits are of high purity, while gray and dark tones are usually caused by carbonaceous matter. In addition, they have a musty or earthy odor, which is caused by their carbonaceous matter content[5].

Titanium dioxide is presented as a white, shiny, and opaque material, which is produced by industrially oxidizing titanium minerals such as ilmenite at high temperatures. Its chemical formula is TiO_2. Titanium dioxide is a chemical substance that is used more than titanium metal, this is because titanium dioxide can be chemically used as a raw material in the manufacture of many products used in daily life[6].

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The calcium titanate compound has the chemical formula $CaTiO_3$, it is a semiconductor material with a perovskite-type structure, this is cubic where the calcium atoms are arranged in the corners, the titanium atom is located at the center of the cube and the oxygen atoms are at the center of each of the 6 faces. From these faces, they form octahedrons of the TiO_6 type[7]. $CaTiO_3$ can be found in nature. Among its characteristics is the chemical formula that follows the ABO_3 pattern, where A and B are cations of different sizes and O are oxygen anions. Cation A is a large cation, usually alkaline, alkaline earth, or lanthanide. While B is a medium-sized cation, with octahedral coordination and a transition metal[7, 8].

$CaTiO_3$ has generally been synthesized by solid-state reaction between $CaCO_3$ and TiO_2 at high temperatures or prepared by mechanochemical methods as reported by Berbenni *et al.* (2004)[9], Wang *et al*. (2007)[10], and Palaniandy (2009)[11]. These methods are time-consuming, energy-intensive, and produce powders of uniform composition and low quality because all solid-state reactions are topotactic as cited by Kaciulis *et al.* (1998)[12], Park *et al*. (2010)[13], Gralik (2014)[14] and Portia (2018)[15].

Studies on the synthesis of calcium titanate using limestone and combustion methods are scarce, but a work done by Rongsawat in 2019 performed a similar process[16]. It was synthesized by combustion from raw minerals (limestone and calcite), anatase titanium dioxide (A-TiO_2), and magnesium (Mg). The syntheses were divided into two reagent systems (limestone/A-TiO_2/Mg) and calcite/ATiO_2/Mg. The minerals and A-TiO_2 were ground at high energy for 30 min and then mixed separately with Mg by grinding. The products after combustion were leached with 2 M HCl solution to remove impurities. XRD results showed that the leached products of both reagent systems mainly contained $CaTiO_3$.

Seiyisi *et al*., recently started in 2025[17] that one kind of solar cell that makes use of a perovskite-structured material is gaining attention. As perovskite solar cells have been developed between 2009 and 2024, remarkable power conversion efficiency between 3.8% and 26.1% has been documented. Perovskite solar cells therefore have much potential to be the next generation of reasonably priced and efficient photovoltaic solar cell technology. Additionally, because perovskite solar cells are manufactured from easily accessible and reasonably priced basic materials, they have recently acquired popularity and offered a great economic opportunity. Perovskite solar cells are still in their infancy as far as commercial manufacture and use are concerned. As a result, considerable efforts are being made to advance the creation of industrial production lines and the scalable fabrication of perovskite solar cells.

One of the bases of the history of the state of Coahuila is mining, it can be said that since the colonial era the minerals that have formed the edges of the structure that supports mining in Coahuila, all have their importance and value[18]. This project addresses the hypothesis that there are alternative routes for obtaining calcium titanate that have not been fully explored, one of which would be through the mineral route. In addition to the objectives, which would be to demonstrate the aforementioned hypothesis, the project seeks to find ways to take advantage of the abundant mineralogical resources of the state of Coahuila, Mexico, to increase the state's technological knowledge and competitiveness.

Methodology

First, a thermodynamic study was done using the HSC Chemistry 5.11 software to determine the thermodynamic variables that helped to decide the temperatures used in the process. Using the same HSC Chemistry 5.11 software a Pourbaix diagram was made. Then, the gray limestone mineral from Ejido Dolores belonging to Castaños, Coahuila, Mexico was prepared through the following stages.

Grinding and screening of minerals

The mineral was ground in a BICO Inc. brand ball mill (44.5 lb, 285 Ball Charge, 12 inches x 12 inches, cast iron drum), with a ratio of 1:3 of mineral and balls, in order to obtain better grinding, for a time of 1.5 hours. Once the mineral was ground, it was screened in the Tyler Ro-Tap RX-29 sieve shaker for 20 minutes, where the following aperture numbers were used: 60, 100, 120, 150, and 270. The mesh size used was 270 as the best option.

Mineral washing and X-ray fluorescence

After choosing the 270-mesh size, 10 grams of mineral were weighed on an Explorer Ohaus analytical balance and washed in a 30% water-70% acetic acid solution and 100% acetic acid at a temperature of 60 °C in a hot plate magnetic stirrer (Thermo Fisher Scientific Cimarec) for 15 minutes with magnetic stirring. It was then filtered in a glass cone with filter paper and washed with distilled water. It was left to dry at room temperature until all moisture was removed for 24 hours. Around 1 gram of the washed and unwashed samples was analyzed with the Panalytical brand Epsilon 1 X-ray fluorescence. The Epsilon 1 used a 50-kV silver X-ray tube.

Synthesis of Calcium Titanate ($CaTiO_3$)

For the synthesis of calcium titanate, a stoichiometric ratio of the precursors was used, one of which is a mineral ($CaCO_3$), and the other is titanium oxide TiO_2 nanopowder from Sky-Springs Nanomaterials (TiO_2, rutile, 99.5% purity, 10-30 nm). The molar ratios used were as shown in Table 1. To perform the calcination ramp, the sample was first heated to 100 °C in the muffle furnace for 30 minutes, then gradually reached the temperature indicated in Table 1 and was maintained for 7 hours. Finally, the temperature was slowly lowered over approximately 2 hours until room temperature (25 °C) was reached; this was done to avoid thermal shock. The synthesis reaction was done using a Witeg brand muffle with powder-coated steel body and ceramic fiber insulation, with a capacity of 1200 °C.

Table 1. Experimental conditions (time of 7 hours)

Temperature (°C)	$CaCO_3$ (Mol)	TiO_2 (Mol)	Ratio
600	1	1	1:1
	2	1	2:1
	1	2	1:2
700	1	1	1:1
	2	1	2:1
	1	2	1:2
800	1	1	1:1
	2	1	2:1
	1	2	1:2

X-ray diffraction

The mineral and the products obtained from the calcination reactions were characterized by X-ray diffraction, in a Bruker D8 Advance diffractometer with a scanning range of 10° to 80° on the 2θ scale, with a scanning speed of 0.01 °/s, with CoKα type radiation, the operating conditions were 40 mA and 35 kV. Match 1.1 software was used for phase identification.

Results and discussion

X-ray fluorescence

The grinding was done in the BICO Inc. brand ball mill and the sieving obtained a particle size of 270 mesh, which was suitable for carrying out the calcination reactions of the mineral with titanium dioxide. The objective of having a small particle size is to increase the contact area of the precursors.

The mineral washing process done with 10 grams of mineral and acetic acid at 70 and 100% using a temperature of 60 ºC, indicates that when the concentration of acetic acid increases, a decrease in the calcium percentage occurs, however, it is not so significant. This calcium percentage was determined using the X-ray fluorescence spectrophotometer, which can be seen in Table 2.

The percentage of calcium in the original sample was 65.995%, when treated with a 70% acetic acid solution at 60 °C for 15 minutes, its percentage decreased to 63.825%, and when treated under the same time and temperature conditions with a 100% acid acetic solution, the percentage of calcium continues to decrease, but not as significantly since a percentage of 63.339% of Ca is reported.

Table 2. Chemical analysis (%) of untreated mineral and mineral treated with acetic acid.

Element	Untreated mineral	With acid (70%)	With acid (100%)
Si	0.337	0.368	0.198
S	0.053	0.062	0.36
Cl	0.225	0.215	0.059
K	0.032	0.037	0.041
Ca	65.995	63.821	63.339
Mn	0.021	0.016	0.016
Fe	0.996	0.519	0.422
Sr	0.089	0.235	0.063
Sn	0.012	0.01	0.012
Ce	0.051	0.074	0.059

Thermodynamic analysis and Pourbaix diagram

Why calcium decreased in small amounts, according to X-ray fluorescence analysis, in the presence of acetic acid, can be explained using the thermodynamic data for the reaction:

$$2CH_3COOH + CaCO_3 = CO_2 + H_2O + Ca(CH_3CO_2)_2 \qquad (1)$$

which are shown in Table 3. The thermodynamic variable free energy ΔG(Kcal), is negative at low temperatures, at a temperature of 70 ºC its free energy is -0.017 Kcal, and at 80 ºC its ΔG(Kcal) is equal to 0.170 Kcal, the sign of the free energy is positive, that is, calcium carbonate ($CaCO_3$) does not decompose into carbon dioxide CO_2(g), water (H_2O) and calcium acetate ($Ca(CH_3CO_2)_2$. With the acid treatment applied to the mineral at 60 ºC, a certain amount of calcium carbonate did react with the acetic acid forming calcium acetate in aqueous ions, which remained in the filtered solution; however, these are considered minimal losses since from 65.995 % of the initial calcium percentage, when washed with acetic acid undiluted in water, a calcium of 63.339 % remained, decreasing by 2.656 %. It was possible to maintain the stability of the compound without any pretreatment or post-treatment; the chemistry and the performance of the reaction itself made it possible not to perform those steps.

Table 3. Thermodynamic data of the reaction 1.

T (°C)	ΔH (Kcal)	ΔS (Cal/K)	ΔG (Kcal)
0.000	-4.236	-12.274	-0.883
10.000	-3.217	-8.514	-0.806
20.000	-3.654	-10.032	-0.713
30.000	-4.113	-11.570	-0.605
40.000	-4.592	-13.125	-0.482
50.000	-5.091	-14.695	-0.343
60.000	-5.611	-16.279	-0.188
70.000	-6.151	-17.874	-0.017
80.000	-6.710	-19.481	0.170
90.000	-7.289	-21.097	0.373
100.000	-7.886	-22.721	0.592

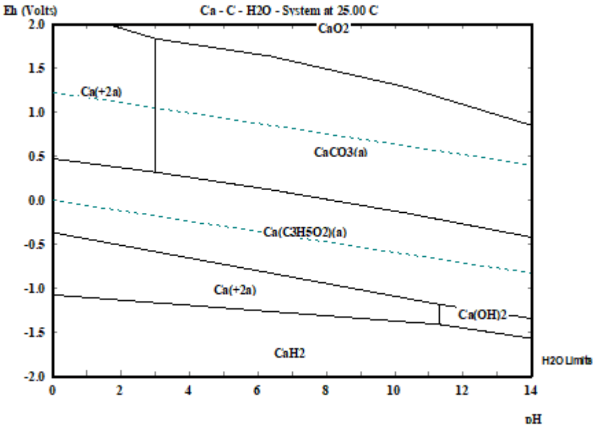

Fig. 1: Eh-Ph system for Ca-C-H_2O.

The Pourbaix diagram representing the potential (ordinate) as a function of pH (abscissa) for a given metal under standard thermodynamic conditions (usually water at 25ºC) is shown in Figure 1. In this figure it can be seen the calcium carbonate phase corresponds to a stable phase because it is in the oxidation area with negative free energies, even at low pH, the calcium carbonate phase is the predominant one. Considering that if what we used was an edible acetic acid, which has the characteristic of being a weak acid, that is, it generates fewer H^+ ions in water.

This means that the largest proportion of an acetic acid solution in water is found in undissociated form (CH_3COOH); while a small portion is found as the conjugate base (CH_3COO^-), accompanied by H^+ ions, or more correctly, H_3O^+ ions. Since the amount of hydrogen released by this weak acid in an aqueous solution is low, the pH it produces is higher (basic) than that produced by a strong acid.

Regarding calcium titanate, the thermodynamic data for the synthesis reaction:

$$CaCO_{3(s)} + TiO_{2(s)} = CaO{*}TiO_{2(S)} + 2CO_{2(g)} \qquad (2)$$

In the temperature range from 0 to 1000 °C they are shown in Table 4. It is a reaction that begins to be possible from 400 °C, where the value of the free energy (ΔG) appears negative (-3.066 Kcal).

Table 4. Thermodynamic data of the reaction 2.

T (°C)	ΔH (Kcal)	ΔS (Cal/K)	ΔG (Kcal)
0	23.203	39.565	12.396
100	23.081	39.202	8.453
200	22.845	38.645	4.561
300	22.575	38.126	0.723
400	22.288	37.665	-3.066
500	21.989	37.251	-6.812
600	21.675	36.87	-10.518
700	21.341	36.508	-14.187
800	20.981	36.156	-17.82
900	20.591	35.809	-21.418
1000	20.208	35.495	-24.983

According to Croker et al.(2009)[19], the following possible reaction might be used to illustrate how TiO_2 (used in this study) and a calcium phase react to generate calcium titanate in alkaline environments.

$$Ca^{2+} + TiO_2 + 2OH^- \rightarrow CaTiO_3 + H_2O \qquad (3)$$

$$Ca(OH)_2 + TiO_2 \rightarrow CaTiO_3 + H_2O \qquad (4)$$

$$CaCO_3 + 2OH^- \rightleftarrows Ca(OH)_2 + CO_2^{3-} \qquad (5)$$

$$CaCO_3 + 2OH^- + TiO_2 \rightleftarrows CaTiO_2 + H_2O + CO_2^{3-} \quad (6)$$

X-ray diffraction

The amount of calcium reported in the X-ray fluorescence analysis is in the form of calcium carbonate, as indicated by the X-ray diffraction analysis in Figure 2, which according to the Match 1.1 software, shows that the phase found in the mineral is calcite ($CaCO_3$) hexagonal structure, with a lattice parameter an equal to 4.99 Aº, and c equal to 17. 0615 Aº. It is observed that almost homogeneously most of the peaks correspond to the calcite phase. This result is consistent with that found by Vélez *et al.* (2014)[20], who synthesized calcium carbonate from eggshell, it is observed that we have a coincidence of peaks corresponding to calcium carbonate at the same angles 2θ.

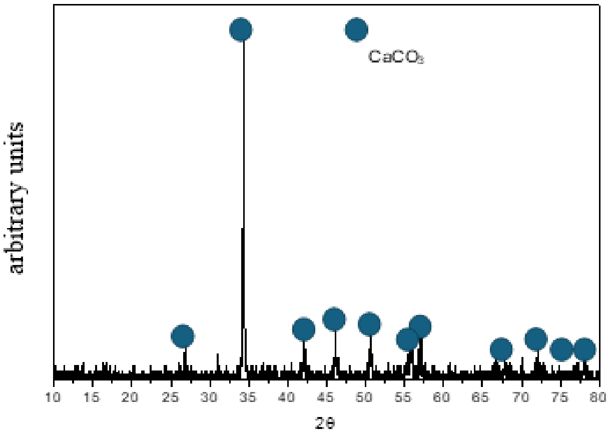

Fig. 2: Diffractogram of the unwashed mineral

It can be said that 70% acetic acid only acted as an impurity cleaning agent without affecting the composition of the mineral, since the limestone phase remains stable, which could be corroborated with the X-ray diffraction technique. The mineral washed with 100% acetic acid analyzed with the X-ray diffractor also shows the calcite phase Figure 3. It can be concluded that acetic acid in high concentrations does not significantly affect the stability of the calcite compound present in the mineral.

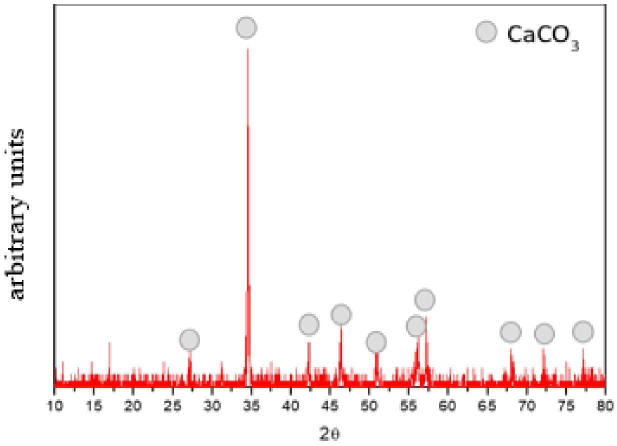

Fig. 3: Diffractogram of the mineral washed with 70% acetic acid

In the first 1:1 molar ratio, which we subjected to temperatures of 600, 700, and 800 ºC for 7 hours, the following was found: at the temperature of 600 ºC, the titanium oxide phase, called anatase, is observed, this compound presents a tetragonal crystalline structure, lattice parameter equal to 3.7850 Å and parameter c equal to 9.5140 Å[21]. The calcite phase is also present, and the calcium titanate phase, $CaTiO_3$, identified with the red dots, is also present, but in small quantities[22]. This phase has a perovskite structure, according to Match software, 1.1[21].

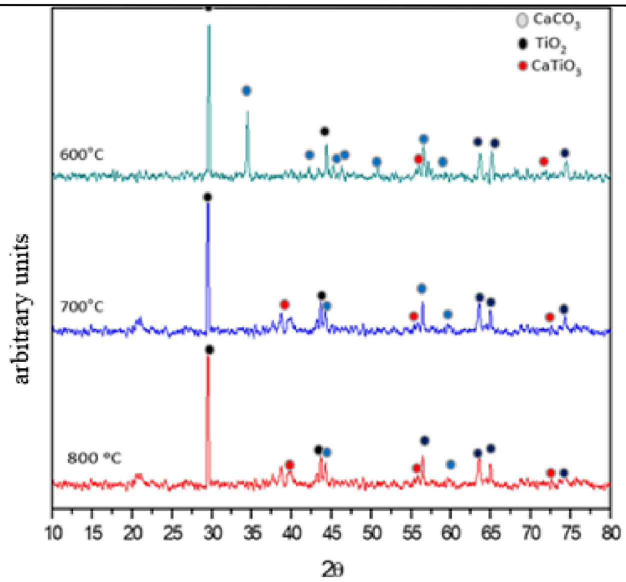

Fig. 4: X-ray diffraction for 1:1 ratio

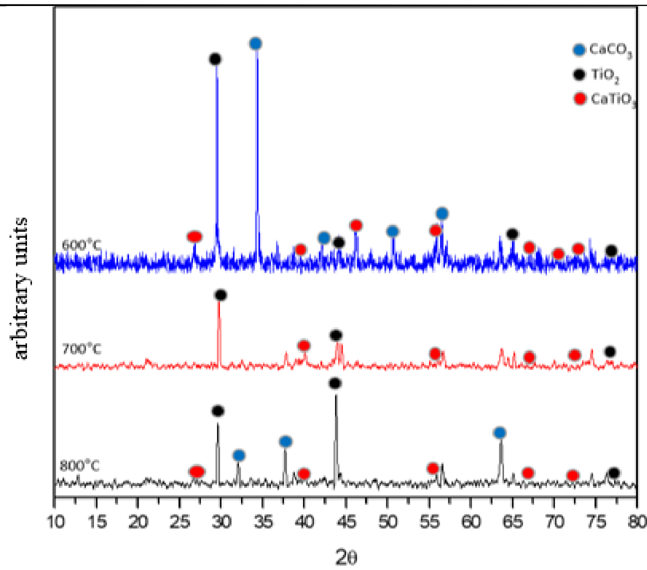

Fig. 5: X-ray diffraction for 2:1 ratio

At a temperature of 700 ºC, it is observed that the calcite phase has decreased and the presence of perovskite increases, however, the amount of this compound is still small. At a temperature of 800 ºC, it practically follows the same pattern as at a temperature of 700 ºC shown in Figure 4. TiO_2 was also detected within the composition of the samples[23].

In Figure 5, the diffractogram of corresponding to a 2:1 ratio, that is an excess of mineral (calcium carbonate) at temperatures of 600, 700, and 800 ºC. In the blue line corresponding to 600 ºC, we observe more peaks corresponding to the perovskite phase ($CaTiO_3$), with an orthorhombic structure with axes equal to 5.302 Å, b equal to 5.3666 and c equal to 5.444 Å, according to Match software, 1.1[21]. This behavior may be because there is more calcium carbonate to react with titanium dioxide, but the three phases still coexist.

At 700 ºC, we did not see the calcite phase, only the anatase and perovskite phases were present. At 800 °C, the calcium carbonate phase was present again, but not in the calcite phase, but in the form of Aragonite with an orthorhombic structure, with network parameters a = 4.96, b = 7.96 and c = 3.77 Å[23]. This phase change from calcite to aragonite could be due to the temperature where the vibration of the atoms increases and a change in the crystalline structure and the atomic arrangement is achieved.

The ratio of 2:1 at 600 ºC, compared to ratio 1:1, achieved better results, because the same temperatures were used but different stoichiometric proportions, it can be thought that this factor of the proportions is the one that has the most impact when performing the synthesis of calcium titanate ($CaTiO_3$). Comparing the findings of this work with other investigations. Lozano, (2014)[7], who synthesized calcium titanate by microwave-assisted hydrothermal method from commercial titanium dioxide and calcium chloride as precursors of titanium and calcium. With these conditions, he found the orthorhombic type of perovskite, as in our case. The positions of the peak intensities coincided with the JCPDS chart 00-022-0153 of orthorhombic $CaTiO_3$.

Hinostroza *et al.* (2024)[25], synthesized calcium titanate as a potential sensor of organic contaminants by traditional method, mixing the precursors in ethanol with magnetic stirring and subsequent heating to 1300 °C. They found the perovskite phase at the 2θ values of 23.27, 26.0, 32.9, 33.1, 37.0, 37.2, 39.0, 40.6, 40.9, 42.6, 44.3, 47.5, 49.0, 53.8, 54.7, 59.0, 59.3, 69.1, 69.6 and 79.3. In our case, we identified the phase at 26.0, 40.6, 43.0, 53.0, 64, 69 and 74 in 2θ, for sample ratio 2:1 at 600 °C. A high coincidence with the results is observed, the difference may be because our precursor is the limestone mineral that contains other compounds.

In Figure 6, the diffractograms of 1:2 ratio. The results do not show the presence of calcium carbonate ($CaCO_3$), which is a good indicator because so little of it was consumed to achieve the synthesis of calcium titanate ($CaTiO_3$). It is observed that the predominant phase is titanium dioxide, which results from the excess of this precursor.

Titanium oxide (TiO_2) and calcium titanate ($CaTiO_3$) are the only two phases present at the 3 synthesis temperatures of 1:2 ratio. As mentioned, it is a good achievement not to find the presence of calcium carbonate ($CaCO_3$), but even so, despite the formation of titanate, the titanium oxide phase (TiO_2) is still present in considerable proportions, which has already mentioned may be due to the excess of titanium oxide added.

One possible way to increase purity would be to pretreat the mineral to improve its purity or to find a precursor closer to the desired characteristics. Two potential paths are being considered to achieve this: one is to subject it to another condition heat treatment to eliminate moisture and some volatile impurities. Another would be leaching with a new reagent capable of removing impurities. This would be pursued in potential future studies.

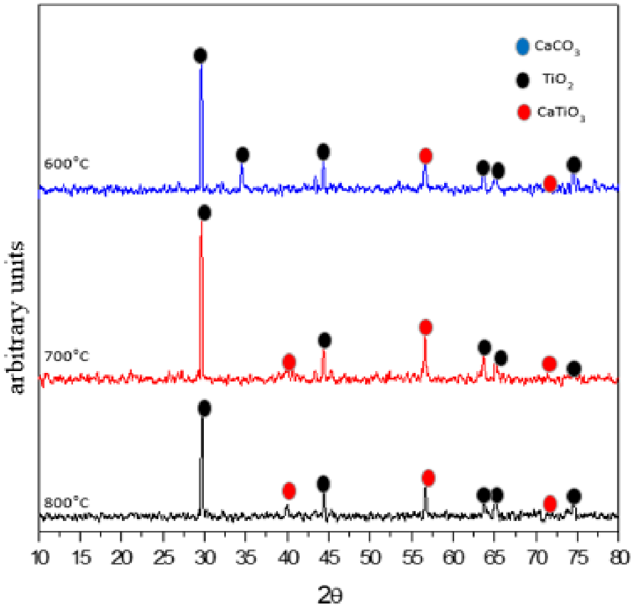

Fig. 6: X-ray diffraction for 1:2 ratio

This investigation, according to the Technology Readiness Level (TRL) [26], is in phase 1 or, at best, in phase 2. The study is in an early phase of development, that is, a basic science, where the goal is to find the best conditions and raw materials to obtain the highest purity of the material. The results observed are encouraging but not yet definitive. It is hoped that with further research and parameter and synthesis adjustments, a product potentially economically exploitable in the market could be obtained.

Conclusions

The mineral ground to a mesh size of 270 is suitable for washing and calcining reactions. The 70% acetic acid, used for washing the mineral, did not significantly affect the decrease in the percentage of calcium and calcium carbonate contained in the mineral. The thermodynamic study for washing the mineral with acetic acid shows that calcium carbonate in a weak acid does not affect its chemical stability. The mineral contains a high content of calcite ($CaCO_3$). The thermodynamic study indicates that the synthesis reaction of titanate $CaCO_3(s) + TiO_2(s) = CaO{*}TiO_2(s) + 2CO_2(g)$, is possible from 400 °C, where the value of free energy (ΔG Kcal) starts to become negative (-3.066 Kcal).

The synthesis of calcium titanate under these conditions occurred but not in a pure way, as indicated by the X-ray diffractograms in the various stoichiometric ratios used. The best synthesis conditions occurred at 600 °C, with a molar ratio of 2 to 1. Compounds such as $CaCO_3$ and TiO_2 were also detected, in some samples the desired compound only occurred partially. A possible way to improve the purity of the material is to subject the mineral to different types of pretreatments. One would be to find new thermal treatment conditions, and another, a new leaching agent. This would be explored in possible subsequent studies. The project is in its early stages according to the Technology Readiness Level (TRL), seeking optimal parameters in basic science, but it is thought that with further research and development, it could become a commercially exploitable product.

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