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Magnesium Titanate Nanoparticles from Dolomite: Cost-Effective Synthesis and Characterization

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Abstract

The production of $MgTiO_3$ regular nanoparticles through the reaction of $MgSO_4$ extracted from dolomite with TiO_2 has been successfully achieved. In this process, the extraction of MgSO4 from dolomite using H_2SO_4 precedes the synthesis of $MgTiO_3$ nanoparticles. To ensure the highest purity of the final product, crucial parameters such as starting materials ratio and calcination temperature were meticulously explored. Through systematic experimentation, the optimal conditions for synthesis were determined to be a 1:1.5 ratio of TiO_2 to $MgSO_4$, coupled with a calcination temperature of 300 °C. This careful optimization strategy yielded MgTiO3 nanoparticles with remarkable regular nanostructures, as evidenced by structural investigations using transmission electron microscopy (TEM). One of the notable advantages of this approach is its energy and cost efficiency. The synthesis process occurs at a relatively low temperature of 300 °C, which not only conserves energy but also suggests its potential applicability in industrial settings. Additionally, the process results in the production of K_2SO_4 as a byproduct, contributing to cost reduction in manufacturing. In conclusion, this study presents a promising method for producing high-quality $MgTiO_3$ nanoparticles with regular nanostructures through a sequence of controlled reactions. The potential industrial viability, low-temperature synthesis, and byproduct generation further highlight the practical significance of this approach in advancing nanomaterial synthesis and contributing to economic efficiency.

1. Introduction

Ceramic nanomaterials are an important platform for researchers in terms of their study and development. The reason for this is due to the fact that most of them have very superior properties in many vital and industrial applications. Tripathy et al., (2016) have proven that the electro-ceramics made of oxide ceramics (ABO_3) are widely employed in an array of uses including insulators, magneto electric, thermoelectric, semi- and transparency, ferro and anti-ferromagnetism, and massive magneto protection [1]. Many methods have been used for the synthesis of the ceramic material such as the chemical co-precipitation and metal organic chemical vapour deposition [2, 3]. One among the significant ceramic industrial materials is magnesium titanates. However, many previous studies have been conducted on the synthesis of the ceramic material ($MgTiO_3$) such as solid state method that includes the mixing of titanium dioxide with a source of magnesium ($MgCO_3$, $Mg(OH)_2$ or MgO) [4-7]. Furthermore, solid-liquid method that includes the mixing of titanium dioxide with a source of magnesium solution such as $Mg(NO_3)_2$ has been used to synthesize of magnesium titanate [8]. Finally, Sol-gel method that includes the using of liquid titanium sources such as ($Ti(OC_3H_7)_4$) with a source of liquid magnesium sources such as ($Mg(OC_2H_5)_2$) [9]. The majority of the reactions in the above mentioned investigations occurred at temperatures above 700 °C [10], implying that getting pure geikielite at a low temperature, which has yet to be accomplished, is an essential target for industry [4-10]. Moreover, from an economic standpoint, the utilization of explicit magnesium sources is costly. As a result, the utilization of natural sources to obtain a magnesium compound is addressed in this work.The nanoform of magnesium titanate has been widely studied since nanomaterials exhibit distinct properties when compared to macro and microstructures of the same material.

However, the studies proved that is difficult to obtain nano $MgTiO_3$ in regular geometric shapes [8, 11-13], therefore, the current study aims to manufacture this material with regular nanostructures and within small sizes compared to previous studies in addition using natural dolomitefor the preparation of the nano $MgTiO_3$.

2. Experimental Procedure

2.1. Materials

All materials were supplied from BioLab and used without further purifications. Dolomite was collected from Al-Ghadari site, Iraq.

2.2. Method

2.2.1. Preparation of $MgSO_4.2H_2O$

Dolomite (125 g) was treated with sulfuric acid 500 ml of 25 wt.% at 60 °C for 30 minutes. The mixture was then centrifuged at 3500 rpm for 10 minutes and the filtered solution was dried on the hotplate and dried in oven at a temperature of 70 °C for 6 hours.

2.2.2. Preparation of $MgTiO_3$ Nanoparticles

TiO_2 (5 g) was added to vigorously stirred HNO_3 (1M, 200 ml) and left for stirring for 15 minutes. Different magnesium sulfate dehydrated ratios were added to the solution with continuous stirring for 15 minutes. The pH of mixture was adjusted to 11-12 by using aqueous solution of potassium hydroxide. The mixture was centrifuged and then the precipitate was washed with distilled water for at least four times to remove potassium sulfate and the isolated samples were dried in an oven for 2 hours. Finally, the product was calcined at various temperatures (300, 500) °C for two hours as shown in Figure (1).

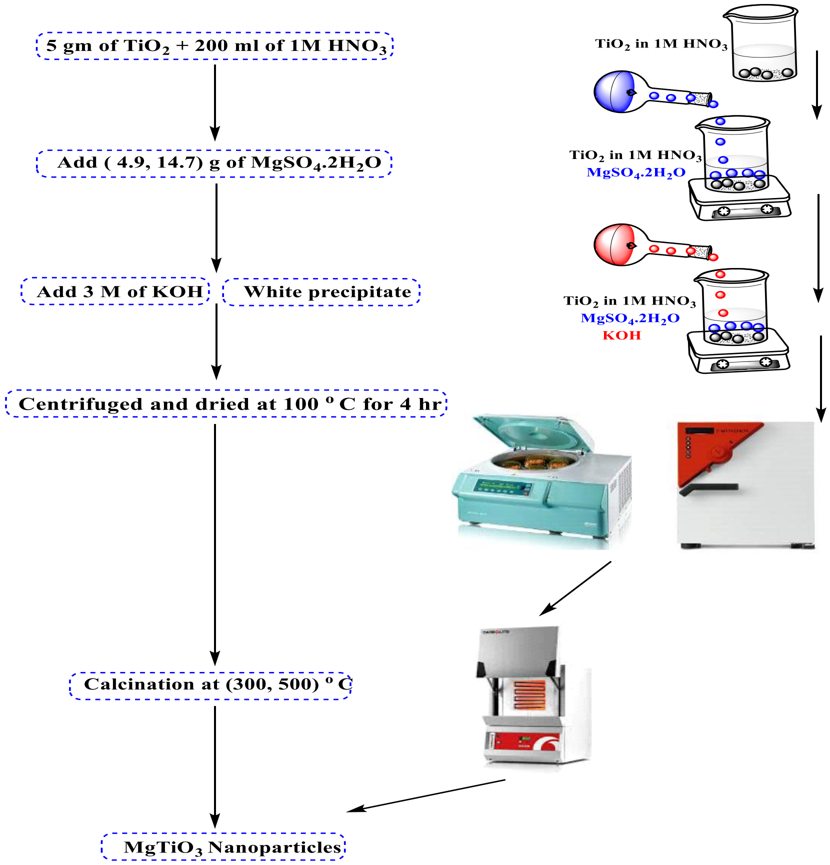

Figure (1). Flowchart of preparation of $MgTiO_3$ nanoparticles.

3. Results and Discussion

During this work, nano-magnesium titanate was produced with a very high purity of up to 99% in an inexpensive way due to the use of a natural source of magnesium, which is dolomite. Furthermore, this work included the studying of calcination temperature and the proportion of the reactants parameters. The resulting material was characterized by using XRD and TEM.

3.1. Effect of Molar Ratio

Two molar ratios were examined in this study; 1:1 and 1:1.5 of Ti: Mg. Depending on the XRD results (Figure 2), both ratios give $MgTiO_3$ but the ratio 1:1 were neglected as it includes the presence of TiO_2 as this starting material could not be able to remove through purification step. The 1: 1.5 ratio was chosen as it is include the formation of geikielite and potassium sulfate which could be removed from the final product by washing after calcination. However, this ratio used is slightly higher or similar to that recorded in previous studies made by [2, 4, 5, 7-9, 14-16].

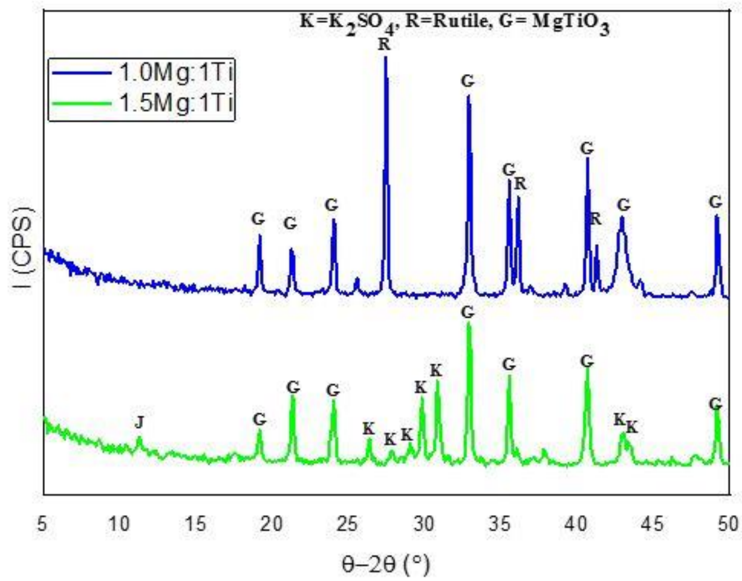

Figure (2). XRD pattern of the molar ratio effect before purification

3.2. Calcination Temperature

The starting materials; TiO_2 and $MgSO_4.2H_2O$ were combined, and the pH was then set at 11-12, followed by an hour of mixing at 25 °C. Thereafter, the mixture was given time to settle, purified before being purified by centrifuge from the aqueous solution, and the precipitate was then dried at 60 °C before being subjected to a two-hour calcination process at 300 and 500 °C (Figure 3). The XRD results showed seven main peaks at 19.26, 21.38, 24.12, 32.99, 35.60, 40.73 and 49.25° of pure $MgTiO_3$ nanoparticles with Miller indices of 003, 101, 012, 104, 110, 113 and 024, respectively. Before performing the calcination process, the mixture of the reactants was washed with hot water in order to remove all sources of potassium from the reaction mixture, thus obtain the final product in a pure form, because the presence of potassium causes the presence of other compounds such as jeppite or the formation of other phases of geickielite. This finding demonstrates that the calcination temperature has no influence on the specific product, and as 300 °C does have the lowest cost, it will be selected as the perfect temperature. Since the minimal degree of calcination heretofore utilized was not less than 600 °C as previously stated, the outcome is therefore regarded as favorable [6-9, 14, 17, 18].

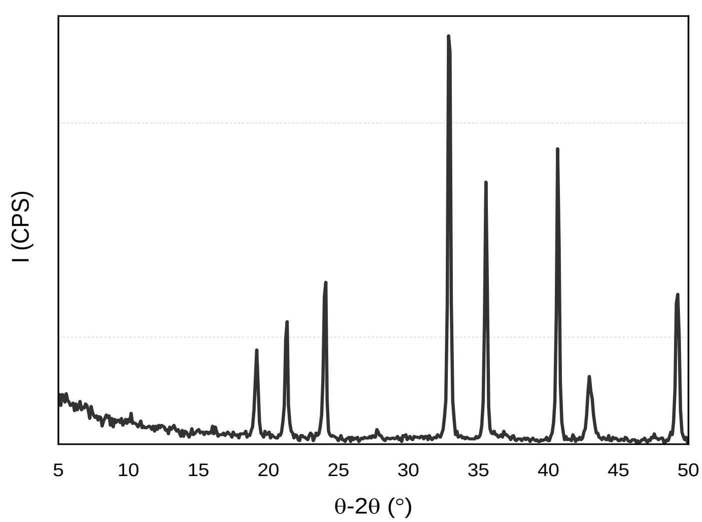

Figure (3). XRD of the $MgTiO_3$ synthesized at 300 and 500 °C.

3.3.1. TEM of $MgTiO_3$

TEM measurement (Figure 4) proves the existence of irregular nano structures of $MgTiO_3$ nanoparticles. The measurement proved that the diameter of these particles reached 30-70 nm, with an average particle size of 48 nm, as shown in (Figure 5). This size is considered the most appropriate size for $MgTiO_3$ nanoparticles was previously prepared. Furthermore, the measurement proved that the MgTiO3particles were well separated from each other, indicating the stability of these $MgTiO_3$ particles.

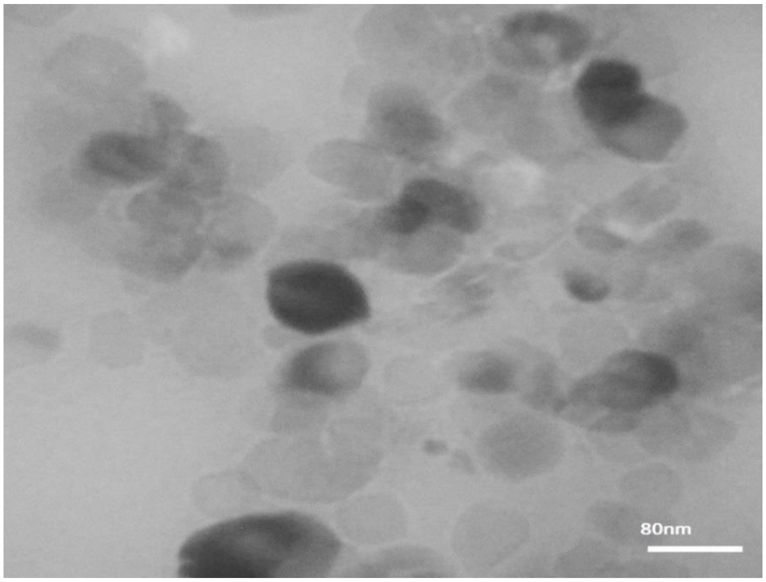

Figure (4). TEM of the $MgTiO_3$ synthesized at 300 °C.

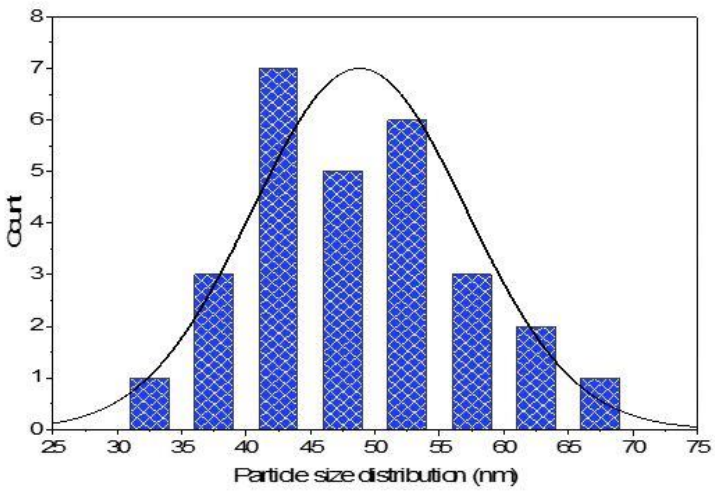

Figure (5). Particle size distribution of the $MgTiO_3$.

4. Conclusions

Using magnesium sulfate from a cost-effective source, dolomite, it was possible to create pure $MgTiO_3$ devoid of other phases. For making nano-$MgTiO_3$ in uniform nanostructures and tiny nanosizes, the technique adopted is thought to be excellent. The technique was cost-effective since it only required a temperature of 300 ºC.

Conflict of Interest: The authors declare that there are no conflicts of interest associated with this research project. We have no financial or personal relationships that could potentially bias our work or influence the interpretation of the results.

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