The effect of synthesis time to particle size of $Bi_4Ti_3O_{12}$ which synthesized using molten single salt NaCl method

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Abstract. $Bi_4Ti_3O_{12}$ was synthesized by the molten salt method using single salt NaCl with calcination time variations: 2, 4, 8, and 16 hours at 900 °C. The effect of time synthesis on particle size and morphology was investigated. The X-ray diffraction data showed that the sample $Bi_4Ti_3O_{12}$ was successfully synthesis and there is not found any impurities. The image from scanning electron microscopy showed that the shape of $Bi_4Ti_3O_{12}$ particle is plate-like and particle size increases as calcination time.

1. Introduction

One of the disadvantages of industrial growth is enhancing water pollutants like dye waste. As a result, it was needed a technology to solve it. Photocatalysis is well known as a potential technology to solve the water pollutant problems due to their ability to degrade the dye waste [1,2]. $Bi_4Ti_3O_{12}$ (BIT) is a member of the three-layered Aurivillius family compound, which recently got attention to be applied as photocatalyst material with the bandgap energy of 2.79 eV (445 nm) [3-5]. Thus, BIT photocatalyst will work in a visible light area. The particle shape/size morphology is one of many factors that influence on photocatalytic activity [6,7]. Previous research reported that the particle shape of BIT is plate-like. Meanwhile, Zhao et al. [4] reported that BIT in micro-scale platelets shape have higher photocatalytic activity in methylene blue degradation. It indicates that controlling a particle morphology is an important factor to enhance the photocatalytic activity.

Molten salt synthesis (MSS) method is a simple synthesis method of materials with an advantage in controllable particle morphology. Chenjie and Zhangsong [8] have synthesized BIT using the MSS method with NaCl-KCl salt at different temperatures (750, 850, 900, 950, and 1050 °C) and reported that BIT was successfully synthesized and plate-like particle formed at 900 °C [8]. In addition, Zhao et al. [9] reported a successfully synthesized BIT plate-like particle using MMS with K_2SO_4/Na_2SO_4 salt [9]. Many synthesis parameters influence the MMS method, such as temperature, salt to oxide ratio, and flux type [10]. Therefore, in this research, we studied the effect of another parameter i.e. time synthesis to molten salt synthesis of BIT, which used NaCl flux.

2. Methods

Bismuth Oxide (Bi_2O_3) and Titanium (IV) Oxide (TiO_2) were ground into mortar agate for one hour in acetone. The sample was put into the alumina crucible and calcined at 700 °C for 6 hours, and NaCl was added with molar ratio of the salt to $Bi_4Ti_3O_{12}$ is 1:7, then grinded in acetone for one hour and calcined at temperature 900 °C with variation time 2 (BIT-2), 4 (BIT-4), 8 (BIT-8) and 16 (BIT-16) hours. The

samples were cooled at 2 °C/min to 500 °C and then the samples were washed using hot deionized water several times until the NaCl content removed.

X-ray diffraction (XRD) data were collected by a diffractometer using Cu-Kα radiation. The range measurement from 2θ(°) = 10–90. The XRD data were compared with Inorganic Crystal Structure Database (ICSD) No. 1559929. The morphology of particle was captured using Scanning Electron Microscopy and the size of the particle was measured using *Image-J* software.

3. Results and discussion

Figure 1 shows the XRD pattern of BIT samples obtained from molten salt synthesis using NaCl salt at variation time synthesis: 2, 4, 8, and 16 hours. By comparing the XRD data sample with XRD data of BIT standard (ICSD no. 1559928) can be known that all peaks of XRD can be indexed to $Bi_4Ti_3O_{12}$ and there is no additional peaks. It means that the samples have no impurities. The morphology of BIT samples was depicted in Figure 2. It can be seen that samples compose many plate-like BIT particles.

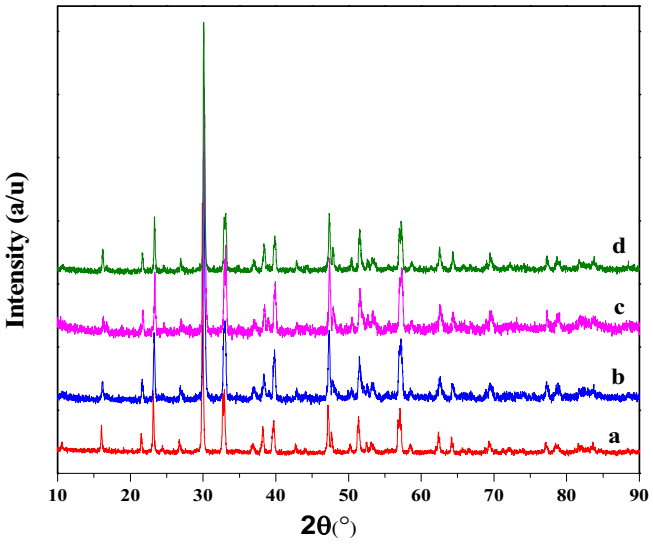

Figure 1. Powder X-Ray Diffraction Pattern of (a) BIT-2, (b) BIT-4, (c) BIT-8, and (d) BIT-16.

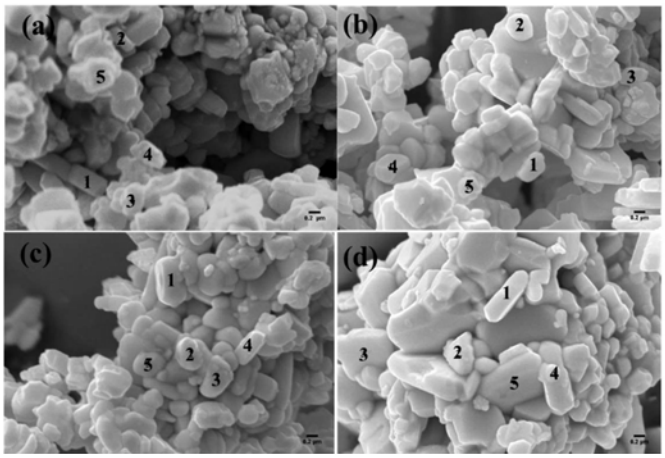

Figure 2. SEM Image of (a) BIT-2, (b) BIT-4, (c) BIT-8, and (d) BIT-16.

SEM image has shown that the samples BIT-2 is smallest and becomes bigger at BIT-4, BIT-8, and BIT-16. Afterward, the size of five different particles was measured using Image-J software, and its results were tabulated in Table 1. Sample BIT-16 has the largest area, among others, and it indicates that the synthesis time corresponds to the particle size. There are two-stage crystal formation mechanisms in MSS i.e., nucleation and crystal growth [9,11]. In this case, the time synthesis corresponds to crystal growth. It can be seen that BIT-16 has the largest area that is caused by the crystal growth phase take more time.

Table 1. The particle size of BIT.

Sample	Particle	Length (μm)	Width (μm)	Area (μm^2)
BIT-2	1	0.687	0.353	0.243
	2	0.503	0.195	0.098
	3	0.595	0.392	0.233
	4	0.629	0.336	0.211
	5	0.672	0.451	0.303
BIT-4	1	0.560	0.304	0.170
	2	0.494	0.423	0.209
	3	0.525	0.296	0.155
	4	0.684	0.511	0.349
	5	0.377	0.377	0.118
BIT-8	1	0,886	0.466	0.395
	2	0,552	0.413	0.228
	3	0,675	0.430	0.290
	4	0,677	0.330	0.220
	5	0,566	0.466	0.264
BIT-16	1	1.045	0.344	0.359
	2	0.645	0.513	0.330
	3	1.021	0.968	0.988
	4	1.264	0.484	0.612
	5	1.252	0.787	0.985

4. Conclusion

The increasing time synthesis in molten salt method influences the particle size of BIT. Larger plate-like BIT particle was formed with more time synthesis, which corresponds to the crystal growth time.

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