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Molten Salt Synthesis of Photocatalyst Material $\text{SrTi}_{x-1}\text{Fe}_x\text{O}_3$ ($x= 0, 0.05, 0.1, 0.15, \text{ and } 0.2$)

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Abstract. SrTiO_3 is a material with perovskite-type structure which received many attentions because it has a wide potential in various fields such as piezoelectric and photocatalyst. The photocatalyst material of SrTiO_3 have band gap energy 3.2 eV, as results it will work on wavelength 387.45 nm (ultraviolet light). Doping metal to SrTiO_3 was reported to decrease its band gap energy. In this research, we synthesized iron-doped SrTiO_3 ($\text{SrTi}_{x-1}\text{Fe}_x\text{O}_3$ ($x= 0, 0.05, 0.1, 0.15, \text{ and } 0.2$)) using molten salt method. The diffractogram of $\text{SrTi}_{x-1}\text{Fe}_x\text{O}_3$ ($x= 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) showed that the samples goal were successfully synthesized, but still found impurities (TiO_2 phase) at $x= 0.2$. The SEM images showed that the morphology of particles is cubic and there are found agglomeration in iron-doped SrTiO_3 . The UV-Vis DRS spectra showed that iron dopant can reveal the light absorption in visible wavelength region.

INTRODUCTION

SrTiO_3 compound is considered as one of the materials with good potential for using in photocatalyst technology with a bandgap energy value of 3.2 eV. Therefore, it only works in regions of light with ultraviolet (UV) wavelength [1]. Many researchers have reported that one of strategy to reduce the bandgap energy is substituting dopants into SrTiO_3 [2-5]. Several metals have been reported as dopants in SrTiO_3 compounds, including Mn, Fe, Co, B, Ag, Pt and Au [2-5]. Da Silva, et al (2014) reported that Fe doped SrTiO_3 with doping variations ($x= 0.05; 0.11; 0.24; 0.50; 0.78; \text{ and } 1.0$) experienced a decreasing of bandgap energy, from 3.2 eV to 1.9 eV [6]. Apart from bandgap energy, particle size, and morphology were reported to influence photocatalytic activity [3,7]. Lai, et al (2015) reported that SrTiO_3 with flower-like particle form has a higher photocatalytic activity than cube and spherical shapes. This phenomenon relates to the higher surface area of the flower-like particle [7].

It is well known that the hydrothermal method and molten salt method (MSS) can produce the unique morphology particle [8]. MSS is a simple method that has several advantages: (a) requiring a relatively low temperature, (b) having short synthesis time, (c) flexible, (d) environmentally friendly, and (e) able to produce materials with unique morphology [9,10]. Li, et al (2010) reported that SrTiO_3 was synthesized by the molten salt method and producing single phase of SrTiO_3 , and there is no impurities were identified [10]. Wang, et al (2019) also reported that SrTiO_3 , which was synthesized by molten chloride salt method at temperature of 974 °C produced a cubic particle morphology [11]. This report shows that MSS can be used for the synthesis of SrTiO_3 material, which has a unique morphology. Therefore, in this study, the synthesis of $\text{SrTi}_{x-1}\text{Fe}_x\text{O}_3$ ($x= 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) was carried out using the molten salt method to obtain Fe doped SrTiO_3 compounds which have typical morphology and having lower bandgap energy.

EXPERIMENTAL

The used precursors were SrCO_3 (Aldrich, 99.9% powder), TiO_2 (Aldrich, 99.9% powder), Fe_2O_3 (Aldrich, 99.9% powder), NaCl (Aldrich, 99.5% powder), acetone, AgNO_3 (Aldrich, 2.5% solution), and distilled water.

The synthesis of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) was carried out by mixing the precursors SrCO_3 , TiO_2 , and Fe_2O_3 , which were adjusted to the stoichiometric calculations. The Homogenized precursors were crushed by using a mortar agate for ± 1 hour and added with acetone. The sample was then calcined at 700°C for 6 hours. Then the sample was cooled at room temperature. After that, the calcined sample was mixed with NaCl with a mole ratio of 1: 7, in the same way, it was crushed and heated at 900°C for 8 hours. The salt (NaCl) contain was removed by wash the sample using hot distilled water until there is no found NaCl .

The obtained samples were characterized using X -ray diffraction (XRD) at the range $2\theta = 20\text{--}80^\circ$ and the XRD data of sample (diffractogram) were indexed to the Joint Committee's standards on Powder Diffraction Standards (JCPDS) for determining the sample phase. The morphology particle and elemental sample analysis were characterized by scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), and the light absorption profile was measured by ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) at wavelength $200\text{--}800$ nm.

RESULTS AND DISCUSSION

The diffractogram of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) was shown in FIGURE 1. (a) and has been matched with JCPDS SrTiO_3 standard data (No. 89-4934). At $x = 0, 0.05, 0.1, \text{ and } 0.15$, it fitted to standard data. In $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0.2$), an impurity of TiO_2 precursor ($2\theta = 25^\circ$) indicates that the reaction between precursors has not been completed. The success of Fe dopant in replacing some of Ti atoms in SrTiO_3 was marked by a shifting as well as a widening of the diffraction peaks at position $2\theta = 32.23^\circ$, which is shown in FIGURE 1. (b), and it can be seen that the greater dopant Fe concentration, the higher peak will shift towards lower 2θ . It indicates a change in lattice parameters, cell volume, and a change in the crystallite size of the sample due to some Ti atoms being replaced by Fe.

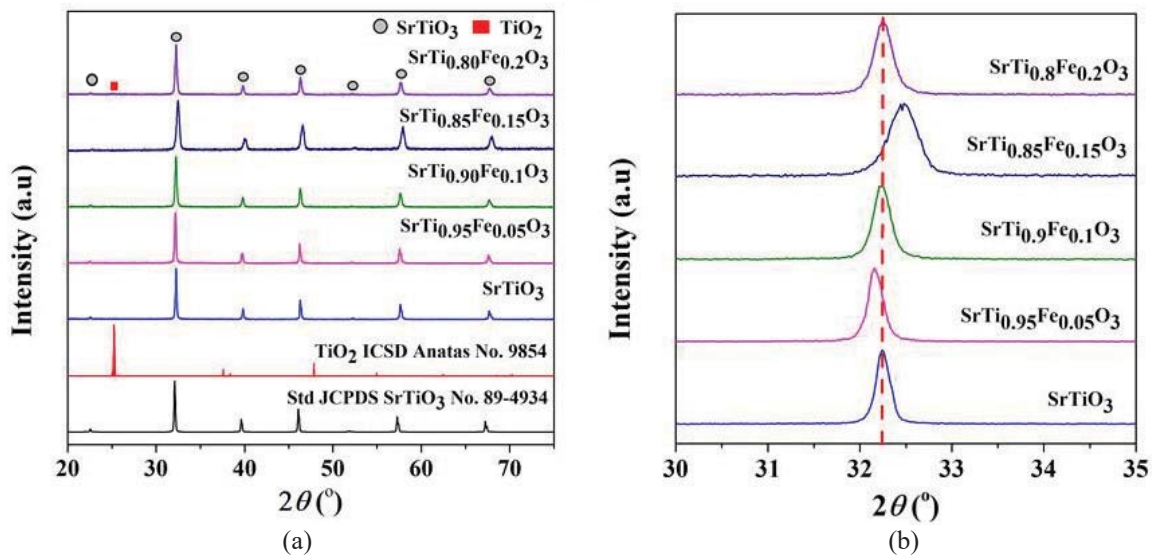


FIGURE 1. (a) The diffractogram of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ compounds ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) and (b) The shift X -ray diffraction peaks of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) at $2\theta = 32.23^\circ$.

The SEM micrograph of sample is shown in FIGURE 2. and it can be seen that the particle morphology of the $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ compounds ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) is cubic. This is the same as that reported by Wang, et al (2019), who synthesized SrTiO_3 using the molten salt method and produced a cubic particle [11]. The scale bar comparison shows that the particle size of Fe doped SrTiO_3 tends to be larger than SrTiO_3 without doping. It showed that the substitution of Fe dopant into SrTiO_3 lattice influenced to particle growth. Besides that the agglomeration formation is possible due to relatively high temperatures [12]. The EDS spectrum is shown in FIGURE 3. and Fe dopant was detected on the sample $x = 0.05, 0.1, 0.15, \text{ and } 0.2$.

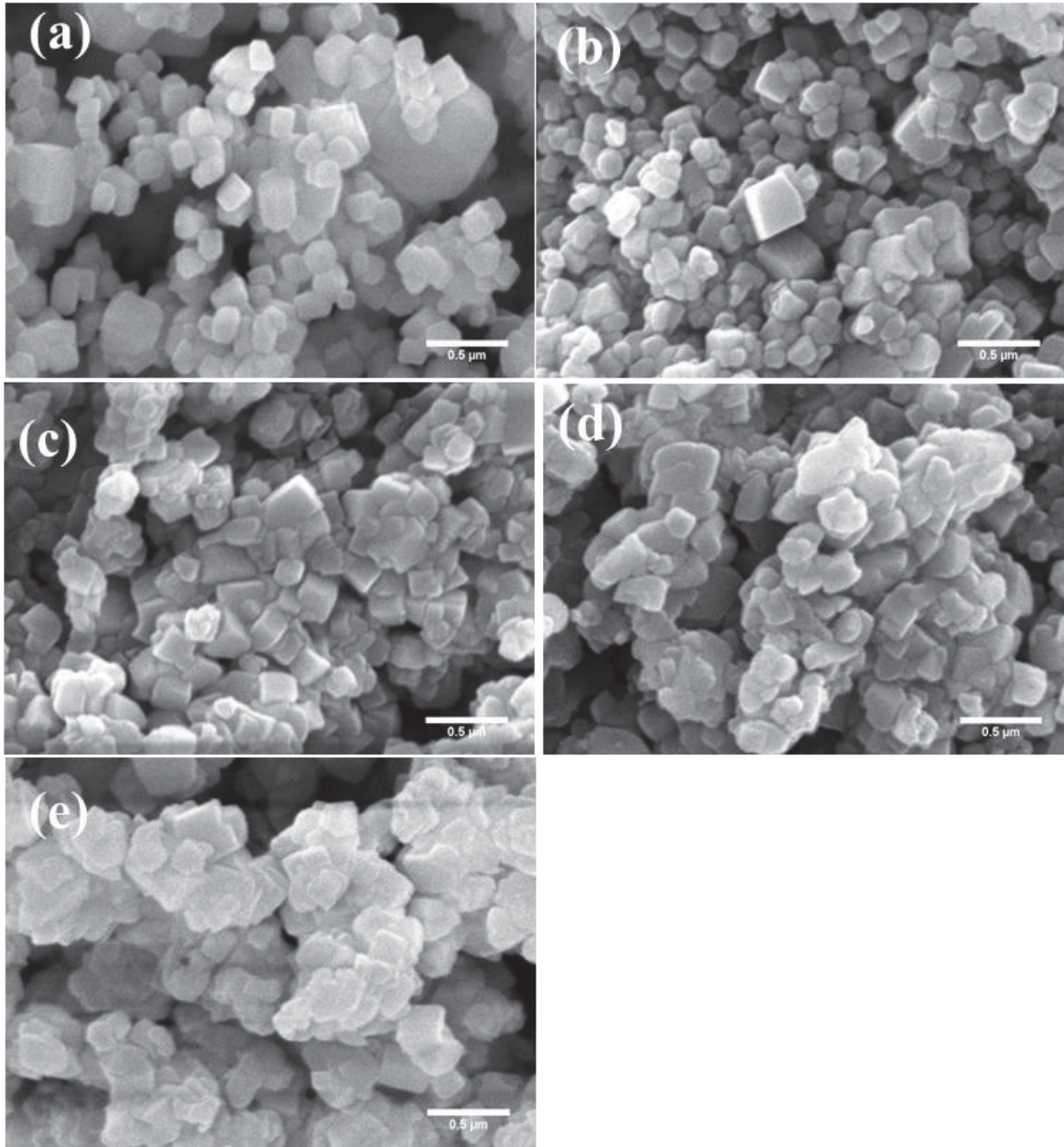


FIGURE 2. SEM micrograph and EDS spectrum of SrTi_{1-x}Fe_xO₃ (a) $x=0$, (b) $x=0.05$, (c) $x=0.1$, (d) $x=0.15$, and (e) $x=0.2$.

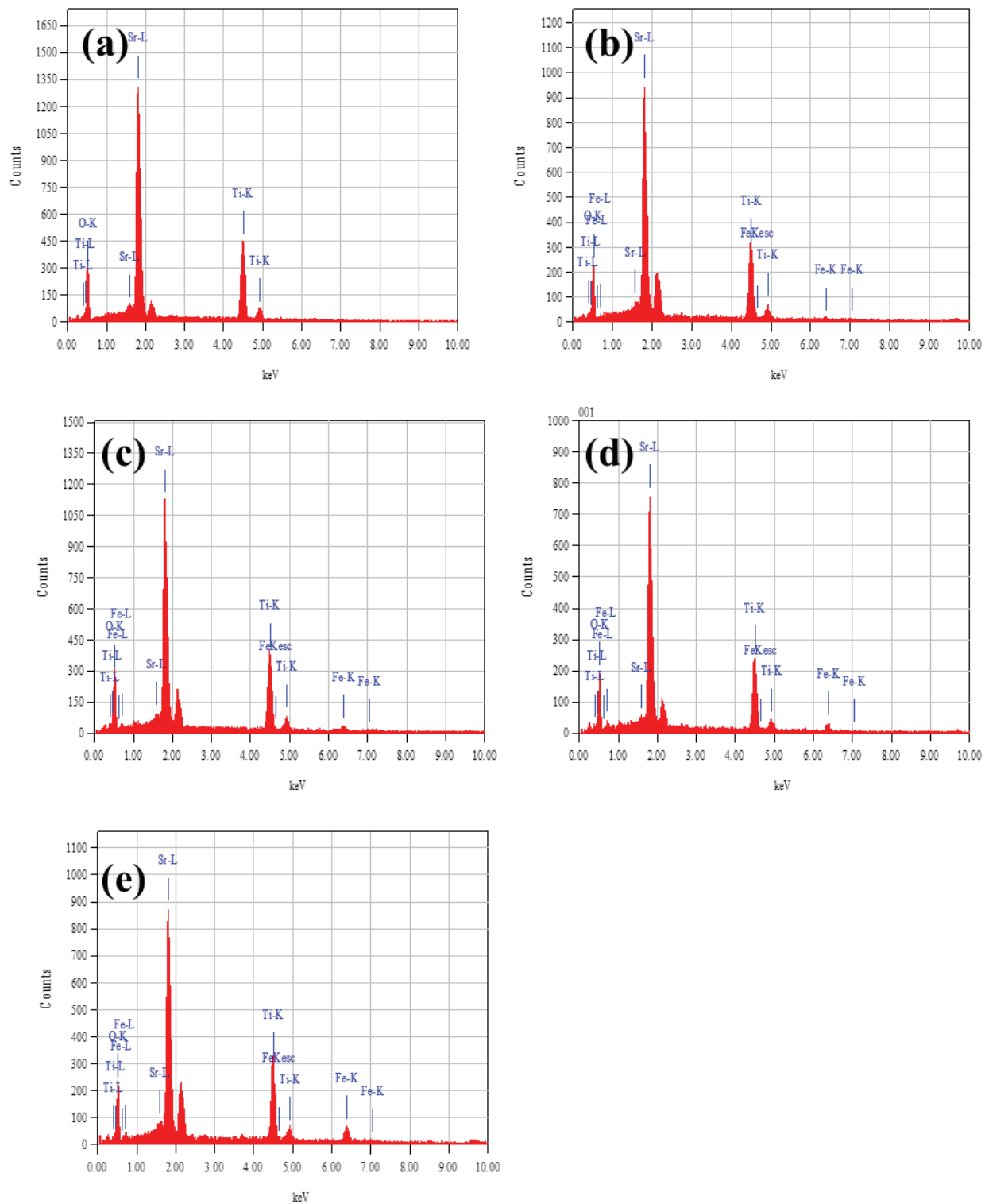


FIGURE 3. EDS spectrum of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ (a) $x=0$, (b) $x=0.05$, (c) $x=0.1$, (d) $x=0.15$, and (e) $x=0.2$.

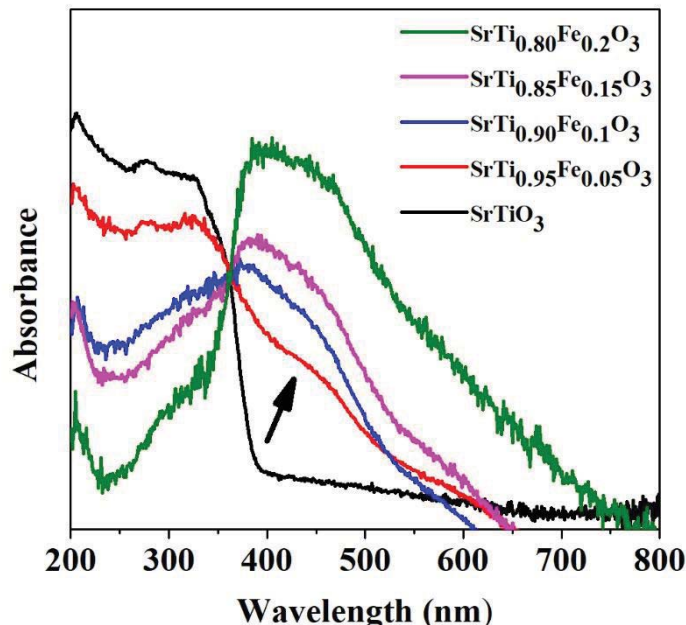


FIGURE 4. DRS spectra of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$).

The UV-Vis DRS spectra $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) are shown in FIGURE 4. It can be observed that SrTiO_3 in the presence of doping changes the character of the absorption pattern where the sample has an ability to absorb light in the visible light area and greater doping concentration of Fe will be higher absorption in that area. It indicates that the presence of Fe dopant in the SrTiO_3 lattice reveal the new state energy level which lies in between valence and conduction band energy of SrTiO_3 [13].

CONCLUSION

Synthesis of the $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) compounds by molten salt method was successfully carried out, but at $x = 0.2$ still found impurities from the precursor of TiO_2 . The particles shape of $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_3$ ($x = 0, 0.05, 0.1, 0.15, \text{ and } 0.2$) is cubic, and the Fe dopant can reveal light absorption in the visible light area.

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