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Research Paper

Molten Salt Synthesis of $SrTi_{0.95}Fe_{0.05}O_3$: The Effect of Chloride Salt Type Study

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Abstract
Strontium titanate ($SrTiO_3$) refers to a perovskite structure compound reported to have photocatalyst properties. It is well-known that modifying the particle morphology is found to be capable of enhancing the photocatalytic activity as it can increase the reaction sites on the solid surface. Molten salt synthesis is one of the compound synthesis methods to produce the homogeneous particle size as well as the unique morphology. One of the parameters determining the product compound obtained using the molten salt method is the type of salt used. In this study, the $SrTi_{0.95}Fe_{0.05}O_3$ was synthesized by the molten salt method using NaCl, KCl, and NaCl/KCl salt. It also studied the effects of chloride salt type to structure, vibration mode, morphology particle, band gap energy and thermal stability of product samples. The diffractograms showed that $SrTi_{0.95}Fe_{0.05}O_3$ were successfully synthesized; however, the sample obtained using molten KCl salt had the impurities of TiO_2, and $SrCO_3$ (residual of precursors) indicating that the KCl flux was insufficient to make a complete reaction. The characteristic of infrared vibration modes of the $SrTiO_3$ compound were found in all samples. The image of scanning electron microscopy showed that all particle morphology was in the quadrate-particle shape, and the $SrTi_{0.95}Fe_{0.05}O_3$-KCl sample had the smallest particles for having the largest surface area. The Kubelka-Munk equation calculation results showed that all samples' band gap energy was approximately at ~3 eV. The DSC curve showed a relatively similar pattern; therefore, the thermal stability properties of all samples were similar.

Keywords
Fe-doped $SrTiO_3$, Molten Salt Method, Chloride Salt Type

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1. INTRODUCTION

The perovskite structure compound has the general formula of ABO_3 with A-site cation as rare earth, alkali, or alkaline earth element (such as: Sr, Ba, Pb, Ca, La) and B-site cation as a transition metal (such as: Co, Cr, Ti, Zr, Nb, Ta) (Assirey, 2019; Dawa and Sajjadi, 2024). They have many interesting properties i.e. photocatalyst, ferroelectricity, piezoelectricity, and high dielectric constant (Wei et al., 2020). As reported, these compounds possess a number of photocatalytic properties, those are $SrTiO_3$, $CaTiO_3$, $BaTiO_3$, $NaTaO_3$, and $AgTaO_3$ (Kanhere and Chen, 2014; Rizwan et al., 2019).

Strontium titanate ($SrTiO_3$) is a perovskite compound with several advantages such as strong chemical resistance and physical stability, high-temperature resistance, easiness to modify the crystal structure through substitution in A/B cation, and superior optical properties (Tanigawa et al., 2017; Sudrajat et al., 2020). As a photocatalyst compound, $SrTiO_3$ has a band gap energy of 3.24 eV enabling it to be active in the ultraviolet region (Li et al., 2007b). The high band gap energy of $SrTiO_3$ however has disadvantages when applied as a photocatalyst material as it requires high photon energy as the electron excitation source. In addition, this compound, as reported, has a high electron (e^-)-hole (h^+) recombination rate that can make its photocatalytic ability reduced and its photocatalytic activity lowered (Ahmadi et al., 2021). Therefore, a strategy is required to overcome it such as doping with metal/nonmetal and making heterojunction structures (Fan et al., 2020; Lee et al., 2023).

The partial small substitution of A or B cation sites (doping) on $SrTiO_3$ can change the electronic structure, thereby increasing the photocatalytic activity (Mohan and Mao, 2020; Xu et al., 2023). Abdi et al. (2020) synthesized $SrTiO_3$ with doping La at the A cation site and Fe at the B cation site, which made the band gap energy decreased as the doping concentration increased and the photocatalytic activity to degrade methyl orange was better than that of undoped-$SrTiO_3$. One of the metal elements reported to have potential as a dopant is Fe due to its ionic radius being similar to that of Ti^{4+}, which allows

it to easily enter the crystal lattice. Xie et al. (2008) synthesized Fe-doped $SrTiO_3$ and reported that the light absorption shifted to the visible region. Meanwhile, Sood et al. (2015) reported that Fe doping to TiO_2 can inhibit its rate of e^--h^+ recombination.

The morphology and particle size are able to determine the photocatalyst activity (Chen et al., 2019; Utomo et al., 2024). Gao et al. (2018) reported that cubic $SrTiO_3$ had a better ability to degrade anionic dyes compared to polygonal $SrTiO_3$. Gao et al. (2018) reported that the cubic $SrTiO_3$ has a good ability to degrade a mixture of rhodamine blue and methylene blue dyes. Similarly, Rahman et al. (2012)) reported that spherical Cu-doped $SrTiO_3$ nanoparticles have an excellent ability to degrade methylene blue. In other words, both morphology and particle size of $SrTiO_3$ affect the ability of its photocatalytic activity.

The synthesis of $SrTiO_3$ material can be carried out through several methods, including hydrothermal, molten salt, solvothermal, solid state, and sol-gel methods (Shen et al., 2016; Putri et al., 2024). The molten salt method is well-known as a method for synthesizing metal oxide compounds that can control the morphology and particle size. Boltersdorf et al. (2015) suggested that the molten salt synthesis method can make it possible for metal oxide compounds to have good photocatalytic ability. Many researchers reported the perovskite compound synthesis using molten salt method such as $BaTiO_3$, $BaZrO_3$, and $SrTiO_3$ (Xue et al., 2018). Several researchers also reported the molten salt synthesis of $SrTiO_3$ as conducted by Putri et al. (2022), Zhang et al. (2016) and Wei et al. (2020) synthesizing nanocubic $SrTiO_3$. In addition, many researchers also reported the metal doped $SrTiO_3$ using molten salt synthesis (Abreu et al., 2016; Murai et al., 2021). Prasetiyo et al. (2021) successfully synthesized the cubic Fe-doped $SrTiO_3$ via molten salt method using NaCl salt and reported that its particle size was large and agglomeration formed. It indicates that molten salt synthesis is able to provide a good opportunity to gain nanocrystal $SrTiO_3$ compound; therefore, it will be advantageous in photocatalyst application. However, the reported work of molten salt synthesis of metal doped $SrTiO_3$, particularly Fe doped $SrTiO_3$ synthesis is so far still limited.

There are many factors determining the morphology of particles obtained using molten salt synthesis including synthesis temperature, synthesis time, salt type, and mol ratio product and salt (Kimura, 2011). Previous researchers reported the influence of salt types on the morphology of $SrTiO_3$ particles obtained through the molten salt method. Kato et al. (2013) reported the molten salt synthesis of $SrTiO_3$ using different salt including LiCl, NaCl, KCl, and $SrCl_2$ and found that the salt type influenced its morphology and particle size. It indicates that the type of salt can affect the morphology of the particles obtained. In addition, the type of salt selected can affect the purity of the final product. It is widely known that chloride salts are chemically inert; therefore, it can be expected to produce a pure product (Gupta and Mao, 2021). It indicates that the use of chloride salts offers advantages in compound synthesis using the molten salt method. On the other hand, the studies of salt type to the molten salt synthesis of metal-doped $SrTiO_3$ are still limited especially about study on the effects of chloride salt types on the obtained product compounds. Therefore, further investigation is required. In this research, we synthesized Fe doped $SrTiO_3$ ($SrTi_{0.95}Fe_{0.05}O_3$) compound using molten salt method with a variety of salt types i.e. NaCl, KCl, and mixtures and studied the effect of salt type on compound product.

2. EXPERIMENTAL SECTION

2.1 Materials

Strontium carbonate ($SrCO_3$, Sigma-Aldrich, 99% powder), titanium dioxide (TiO_2, Sigma-Aldrich, 99.9% powder), ferric oxide (Fe_2O_3, Sigma-Aldrich, 99.9% powder), natrium chloride (NaCl, Merck, 99.9% powder), kalium chloride (KCl, Merck, 99.9% powder), silver nitrate ($AgNO_3$, Merck, 99.9% Powder), and acetone (C_3H_6O, Merck).

2.2 Synthesis

$SrTi_{0.95}Fe_{0.05}O_3$ was prepared using molten salt method with the mol ratio of product and salt is 1:7. The precursor was calculated based upon stoichiometry with the target product of 3 gram. The mixture of precursors and salt was crushed in an agate mortar for 1 hour and added with acetone to make it homogeneous. The sample was then transferred to an alumina crucible and calcined at 830 and 855°C for 6 hours, respectively. Then, to remove residual salt the obtained result was washed with hot distilled water. The identification of salt residue in the sample was carried out by testing the washed filtrate using $AgNO_3$ solution. Finally, the cleaned sample was dried in the oven at 90°C for 3 hours.

2.3 Characterization

In this study, the instrumentations used are presented as follows: (a) X-ray diffraction (Rigaku Miniflex diffractometer, Japan) technique was carried out in the range of 2θ= 3-90° with Cu Kα radiation source (λ = 1.54 Å). The obtained diffractogram of the compound was then compared with the $SrTiO_3$ standard, Inorganic Crystal Structure Database (ICSD) No. 80874, and refined using the Le Bail method on Rietica software. (b) Infrared (IR) vibration modes of the sample were determined using Fourier Transform-IR Spectrometer (Bruker Alpha II, USA), (c) The morphology particle and elemental composition of the sample was identified using the scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) (Jeol JSM-6510, Japan). The morphology and particle size were analyzed using the Image-J software. (d) The surface area of samples were determined using Surface Area Analyzer (Quantachrome Novatouch Lx. Germany). (e) The light absorption property was identified from diffuse reflectance spectroscopy-ultraviolet visible (DRS UV-vis, Thermo Scientific Evolution 220 spectrometer, USA) using the wavelength range of 200-800 nm. The spectrum data were processed by means of the Kubelka-Munk equation to obtain the band gap energy. Here, the Kubelka-Munk calculation used indirect band gap following

the calculation report by Abdi et al. (2020), (f) The thermal analysis was conducted by Differential Scanning Calorimetry (DSC) (Rigaku Thermoplus Evo 2, Japan) at the temperature ranging from 50 to 450°C.

3. RESULTS AND DISCUSSION

Figure 1 shows the diffractograms of $SrTi_{0.95}Fe_{0.05}O_3$ and it can be seen that the diffraction peaks of the synthesized compounds matched with the standard data (ICSD $SrTiO_3$ standard data No. 80874), indicating that the target compounds have been successfully synthesized. The typical peaks of $SrTi_{0.95}Fe_{0.05}O_3$ compounds were found at $2\theta(°)$ = 23; 32.43; 40; 46.62; 52.4; 57.94; 67.92; 72.76, and 77.42. However, the impurities of TiO_2 (2θ (°) = 25.3) and $SrCO_3$ (2θ (°) = 25.8, and 41.5) was still found in the synthesis product using KCl salt, indicating that the reaction was incomplete. In the molten salt synthesis, the process of forming Fe-doped $SrTiO_3$ compounds goes through several mechanisms: dissolution, diffusion, and precursors reacting with each other in molten salt (Li et al., 2007a; Liu et al., 2009). At the diffusion

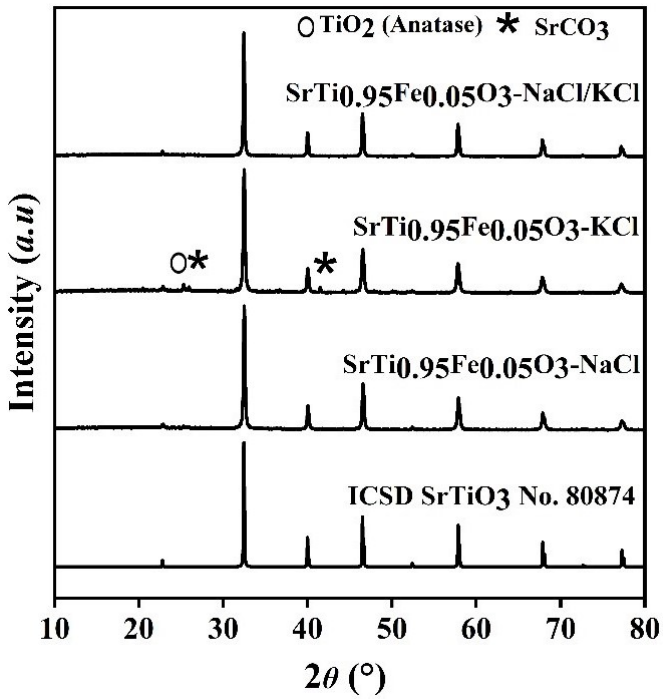

Figure 1. The Diffractograms of $SrTi_{0.95}Fe_{0.05}O_3$ Compounds Synthesized Using the Different Types of Chloride Salt

stage, the reactions of precursors formed product; therefore, the remaining precursors (TiO_2, and $SrCO_3$) as impurities indicated the incomplete reaction. Figure 1 also shows that the diffraction peaks of all samples were high and sharp, which indicated that the salt type did not affect the sample's crystallinity. The Fe-doping in $SrTiO_3$ compounds caused the shift of diffraction peak at $2\theta(°)$ = 32.43 towards a larger position (Figure 2) related to the lattice size change as a result of replacing a small partially of Ti^{4+} (ionic radii (r) = 0.605 Å) metal by Fe^{3+} (r= 0.585 Å) (Abbas and Jamil, 2016; Fuentes et al., 2015).

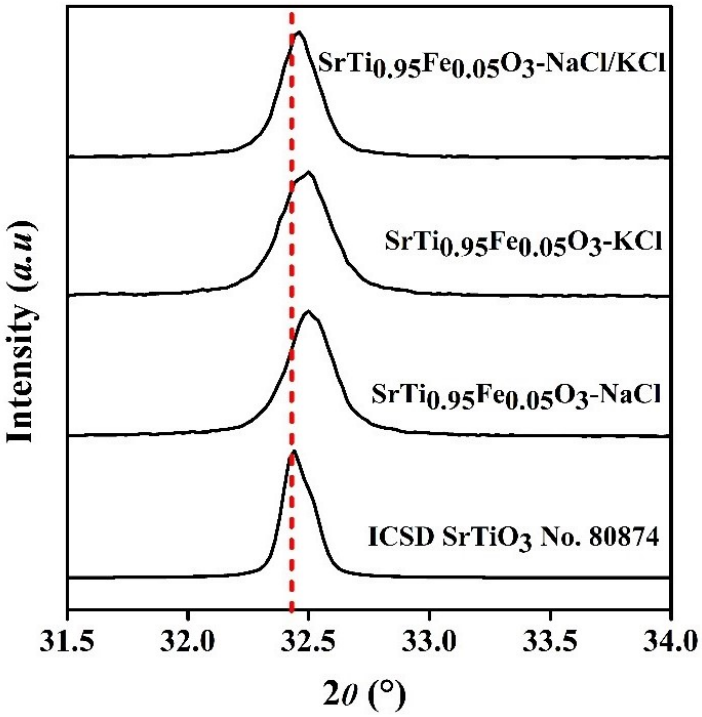

Figure 2. The X-ray Diffraction Peaks Shifting at An Angle of 2θ (°) = 32.43 of $SrTi_{0.95}Fe_{0.05}O_3$ Compounds Synthesized using Different Type of Chloride Salt

The diffractograms of compounds synthesized using NaCl and NaCl/KCl salt were refined by means of Rietica software with the Le-Bail method. The refinement process used the standard of $SrTiO_3$ data on ICSD No. 80874, and the plots of the refinement are shown in Figure 3. The results as summarized in Table 1 shows that the profile residual (Rp) and profile weighted residues (Rwp) values were lower than 15 that indicated the sample diffractograms had good agreement with the standard (Toby, 2006; Nunocha et al., 2022).

Figure 4 shows the IR spectra of the samples in which all of these samples had similar vibrational peaks, i.e. (a) 3200 cm^{-1} relating to OH stretching vibration, (b) 1600 cm^{-1} relating to Ti−O stretching vibration, (c) 1400 cm^{-1} relating to Sr−Ti−O stretching vibration, and (d) 620 cm^{-1} relating to Sr−Ti stretching vibration (Rajkoomar et al., 2020). The OH vibration in all samples indicated the ability of all samples to absorb water. The ability of perovskite to absorb water has been widely reported by previous researchers (Evarestov et al., 2007). A weak vibration peak in sample $SrTi_{0.95}Fe_{0.05}O_3$−KCl was found at 1700 cm^{-1} and corresponded to the vibration of CO_3^{2-} and related to an impurity in sample $SrTi_{0.95}Fe_{0.05}O_3$-KCl ($SrCO_3$) (Zheng et al., 2019).

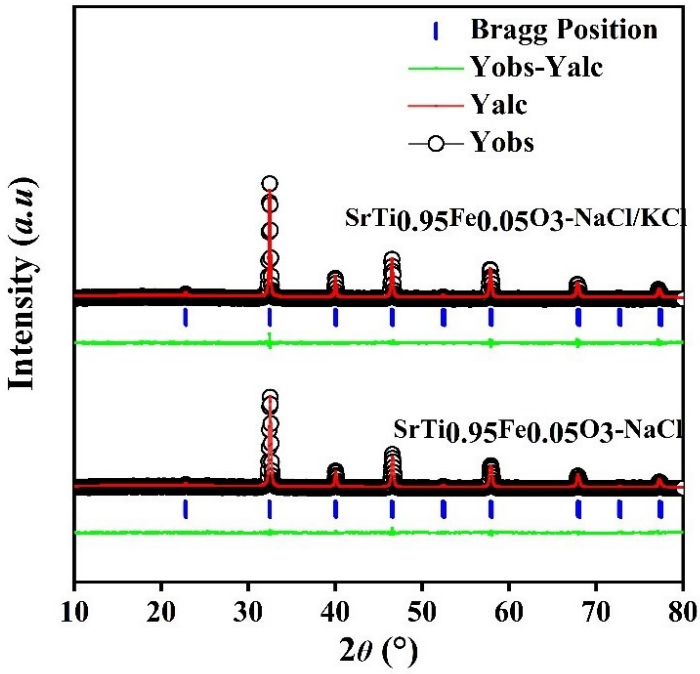

Figure 3. Plots of The Refinement Process Result Using the Le Bail Method for $SrTi_{0.95}Fe_{0.05}O_3$ Compounds Resulting From the Synthesis of NaCl and NaCl/KCl salts

Table 1. The Crystallographic Data of $SrTi_{0.95}Fe_{0.05}O_3$ Synthesized with NaCl and NaCl/KCl Salts Obtained by Refinement Process with Le Bail Method

Parameters	NaCl	NaCl-KCl
Crystal system	Cubic	Cubic
Space group	P*m3m*	*Pm3m*
Asymmetric unit (Z)	1	1
a=b=c (Å)	3.9009 (2)	3.9009 (5)
α, β, γ (°)	90, 90, 90	90, 90, 90
Cell volume (Å3)	59.362 (6)	59.363 (1)
R*p* (%)	8.93	9.13
R*wp* (%)	5.70	6.37
G*oF* (X^2)	0.724	1.090

Table 2. Particle Size Average of Products

Compound	Particle Size Average (μm)
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl	0.2196
$SrTi_{0.95}Fe_{0.05}O_3$-KCl	0.2152
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl/KCl	0.2557

Figure 5 shows the morphologies and particle size distributions of $SrTiO_3$ and Table 2 presents the summary of the calculation results of particle size average. The particle morphology is quadrate-particle shape similar to the work reported by Li et al. (2021) that obtained nano quadrate $SrTiO_3$ particles synthesized using NaCl/KCl molten salt method. The SEM images also showed the similar morphology produced from the use of different salt types; this indicated that the chloride salt type did not affect its morphology particle. On the other hand, the study reported by Prasetiyo et al. (2021) obtained the different morphology that gained Fe-doped $SrTiO_3$ in cuboidal-shape using NaCl salt as the flux. This difference was probably related to the differences in synthesis temperature in which the work by Prasetiyo et al. (2021) used a higher temperature (900°C) leading to the maximum particle growth.

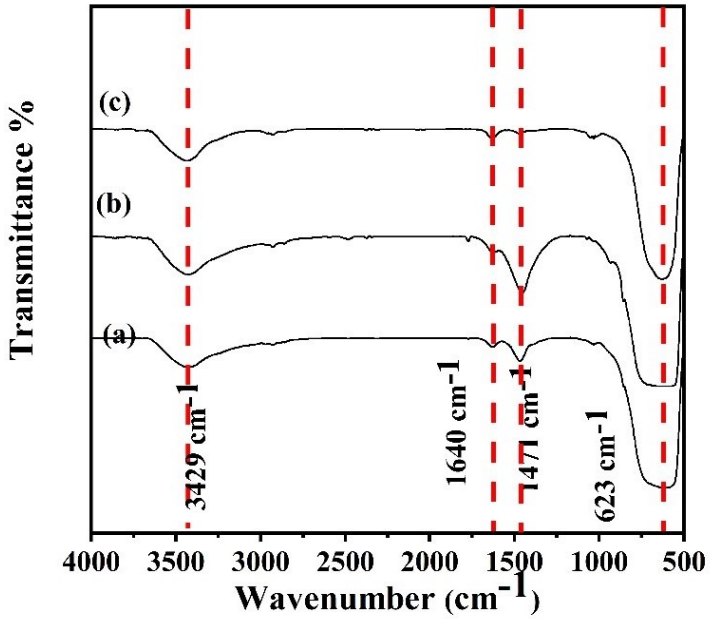

Figure 4. IR Spectra of (a) $SrTi_{0.95}Fe_{0.05}O_3$-NaCl (b) $SrTi_{0.95}Fe_{0.05}O_3$-KCl, (c) $SrTi_{0.95}Fe_{0.05}O_3$-NaCl/KCl

Table 3. Surface Area of Products

Compound	Surface Area (m^2/g)
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl	5.330
$SrTi_{0.95}Fe_{0.05}O_3$-KCl	8.300
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl/KCl	2.466

The results of average particle size (Table 2) showed that the obtained particle size was relatively small as well as similar to 0.2196-0.2557 μm. It indicated that the nucleation rate was higher than the growth rate of the particles so that a large number of particles were obtained but with a small size. The particle size of sample synthesized using NaCl/KCl salt was found larger than others. It was in view of the lowest melting point of NaCl/KCl (657°C) making the particle growth occurred earlier and leading to the larger particle growth. Besides, the physical properties of salt such as melting point, viscosity,

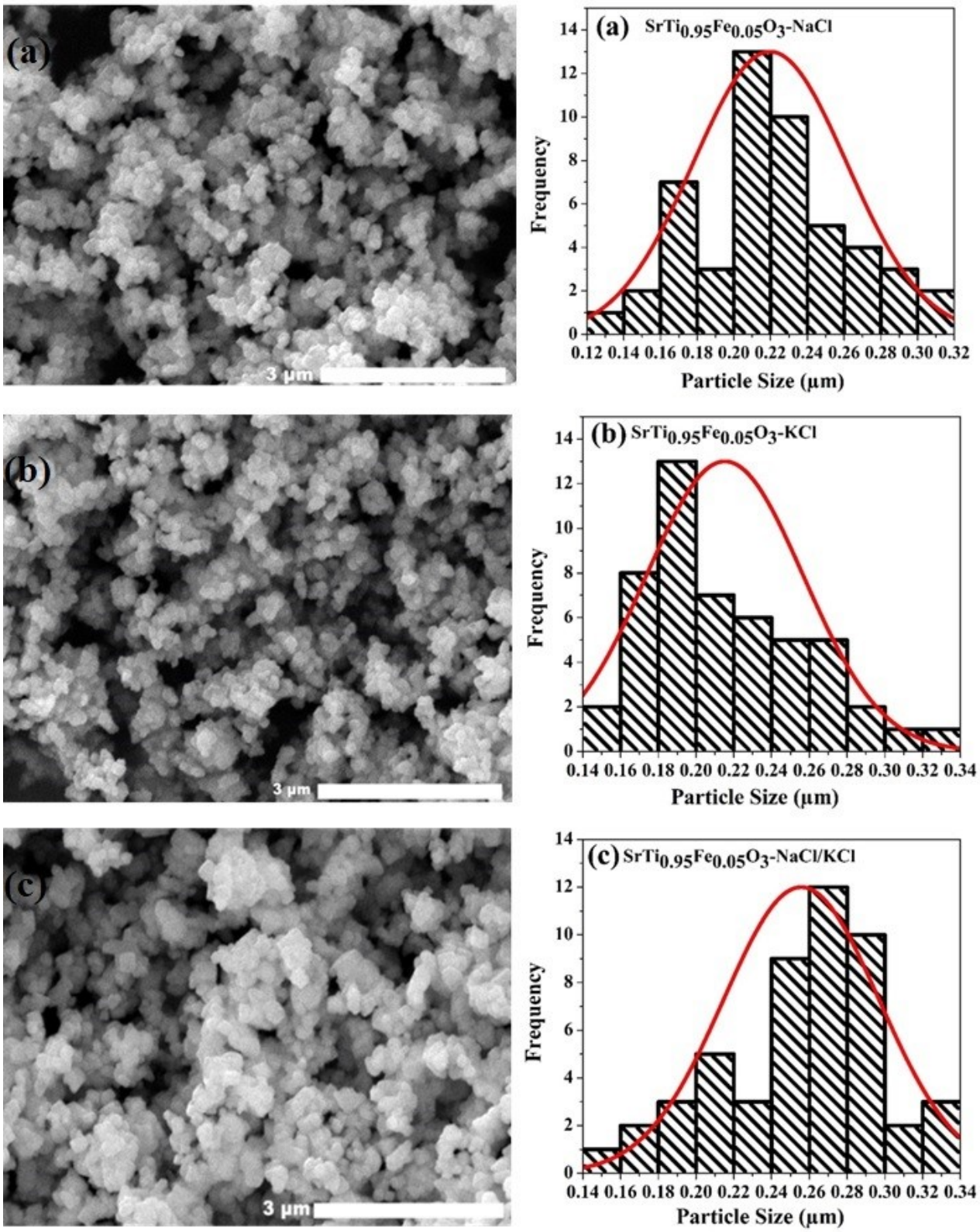

Figure 5. SEM Images and Particle Size Distributions of $SrTi_{0.95}Fe_{0.05}O_3$ Synthesized using (a) NaCl, (b) KCl, and (c) NaCl/KCl Salt

Table 4. The Percentage of Element Mass in $SrTi_{0.95}Fe_{0.05}O_3$

Element	$SrTi_{0.95}Fe_{0.05}O_3$-NaCl (% mass)	$SSrTi_{0.95}Fe_{0.05}O_3$-KCl (% mass)	$SrTi_{0.95}Fe_{0.05}O_3$-NaCl/KCl (% mass)
Stronsium (Sr)	41.00	38.47	41.71
Titanium (Ti)	29.59	27.64	29.49
Iron (Fe)	3.62	3.71	3.44
Oxygen (O)	25.79	30.18	25.36

and solubility can affect the morphology and particle size (Liu et al., 2020). At this point, the melting point of salt affects the formation of particles. The faster the melting point of salt, the quicker the formation of crystal seeds (nucleation stage) (Kimura, 2011). In addition, the viscosity of the salt affects the formation of particles. It corresponds to the lower viscosity induces faster diffusion rate of anions and cations precursors (Li et al., 2007b). Wakao et al. (1991) reported that NaCl and KCl had almost the same viscosity at the same temperature. Therefore, it is possible to obtain the relatively similar morphology and particle size. Table 3 presents the summary of the results of the surface area calculations for the samples in which it can be seen that sample $SrTi_{0.95}Fe_{0.05}O_3$-KCl had the largest surface area. It corresponds to the particle size distribution data that sample $SrTi_{0.95}Fe_{0.05}O_3$-KCl has the smallest particle size (Bullard et al., 2021). Meanwhile, the EDS spectra results (Table 4) showed that the elements contained in $SrTi_{0.95}Fe_{0.05}O_3$ were strontium, titanium, iron, and oxygen indicating that the samples were successfully doped by Fe.

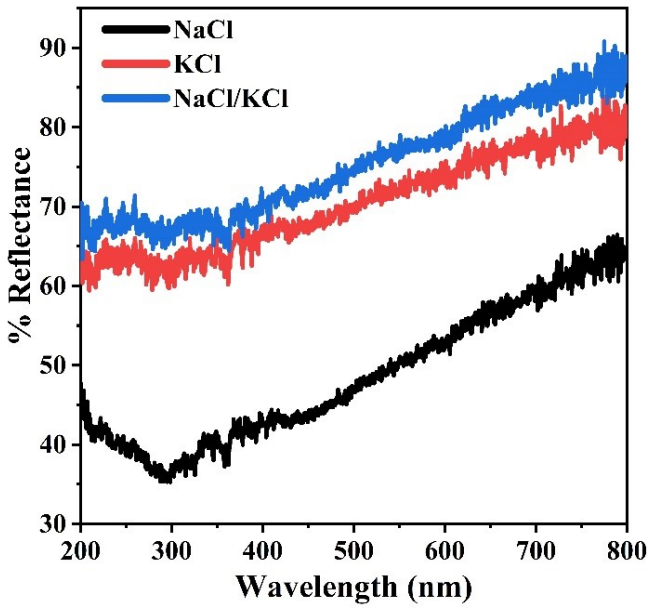

Figure 6. The Percentage of Reflectance Value Vs. The Wavelength of DRS Spectra of $SrTi_{0.95}Fe_{0.05}O_3$ Compounds

Figure 6 shows the reflectance spectra of $SrTi_{0.95}Fe_{0.05}O_3$ and it can be seen that the percentage of reflectance of the sample synthesized using NaCl salt was found less than others, which also showed their higher percentage of absorbance. The difference of percentage of reflectance was probably due to the agglomeration or aggregation particle determining the optical absorption (Melcher et al., 2017). The SEM images (Figure 5) showed that the agglomeration was found in all samples. The Kubelka-Munk equation processed the percentage of reflectance to obtain the band gap energy value. Figures 7 shows the Tauc Plots of $SrTi_{0.95}Fe_{0.05}O_3$ and Table 5 depicts the results of band gap energy calculations. The results of calculation showed that all samples had the relatively similar band gap energy values. The band gap energy of $SrTi_{0.95}Fe_{0.05}O_3$ compound had a lower value compared to the pure $SrTiO_3$ compound (3.24 eV) (Li et al., 2021). It was due to the formation of a new sub band, i.e. d orbital from Fe doping (Abbas and Jamil, 2016). The formation of this new sub band gave the change of the electronic transition from O-2*p* (VB) to Ti-*d* (CB) becomes O-2*p* (VB) to Fe/Ti−*d* (CB), narrowing the band gap energy (Shafique et al., 2021).

Table 5. The Band Gap Energies and Wavelength Absorptions of $SrTi_{0.95}Fe_{0.05}O_3$

Compound	Band Gap Energy (eV)	Wavelength (nm)
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl	3.01	412
$SrTi_{0.95}Fe_{0.05}O_3$-KCl	3.00	413
$SrTi_{0.95}Fe_{0.05}O_3$-NaCl/KCl	2.97	417

Figure 8 shows the thermogram DSC curve of samples and it can be seen that all samples had the similar pattern and no exothermic or endothermic changes were observed. It indicated that all samples had the similar thermal stability trend at the temperature ranging from 50-450°C. The DSC curve also showed that there was no phase transition at 50-450°C. The phase transition of $SrTiO_3$ was not detected due to the short measurement temperature range, while the transition phase occurred outside of this range. $SrTiO_3$ was reported to have a phase transition temperature (structural change from cubic with space group P*m3m* to tetragonal with space group

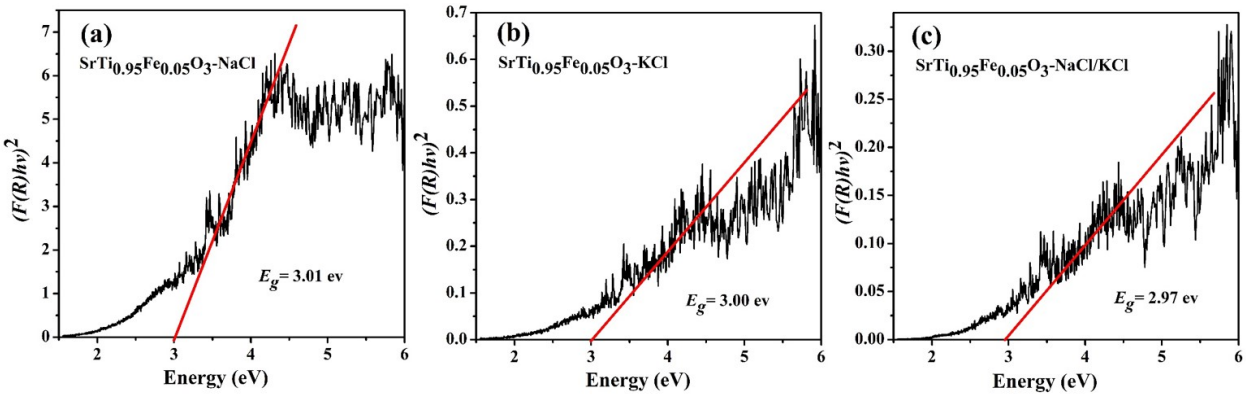

Figure 7. The Tauc Plots of $SrTi_{0.95}Fe_{0.05}O_3$ Synthesized using (a) NaCl (b) KCl, and (c) NaCl/KCl Salts

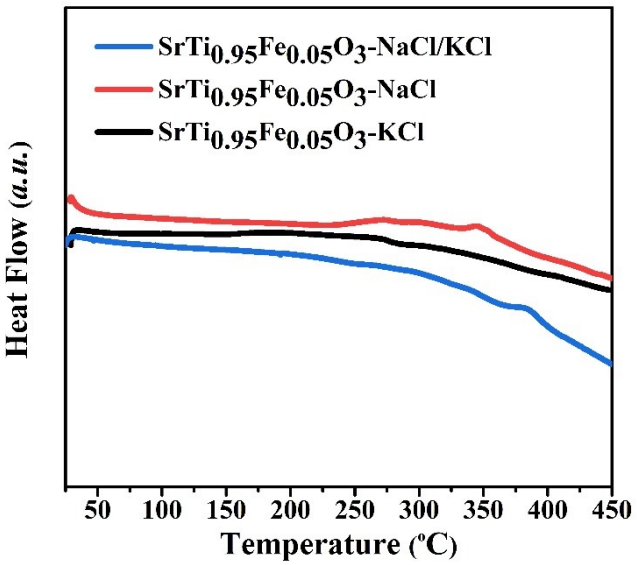

Figure 8. DSC Curve of Samples

P*4mm*) at 105 K (-168.15°C) and melts at 2080°C (Phoon et al., 2019).

4. CONCLUSIONS

The $SrTi_{0.95}Fe_{0.05}O_3$ compounds have been successfully synthesized using the molten salt method with the salt types of NaCl, KCl, and NaCl/KCl. However, the impurities compounds (TiO_2 and $SrCO_3$) were still found in sample $SrTi_{0.95}Fe_{0.05}O_3$−KCl, which indicated that the KCl salt melt has not been able to fully facilitate the reaction of the precursors. The IR spectra of all samples showed the characteristic vibrations of the $SrTiO_3$ compound observed at a peak at the wavenumber 1400 cm^{-1}, which related to Sr−Ti−O stretching vibration, and at a wavenumber of 620 cm^{-1}, which related to Sr−Ti stretching vibration. The obtained morphology was a quadrate-shape with relatively similar particle size. It indicated that the particle growth mechanism of $SrTi_{0.95}Fe_{0.05}O_3$ was similar for all types of chloride salts used. All samples had the relatively similar band gap energy (~3.00 eV/~413 nm) and lower than undoped related to the new formation electronic transition due to Fe dopant. The obtained band gap energy values indicate that this compound offers advantages for photocatalyst applications as it can operate in the visible light spectrum (purple color). Meanwhile, the thermal analysis showed that all samples were similar in thermal stability. The research results indicated that the molten salt method (using NaCl and NaCl/KCl salts) can be used to synthesize Fe-doped $SrTiO_3$ compounds with high purity and uniform small particle size.

5. ACKNOWLEDGEMENT

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