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A simple synthesis and characterization of LaMO$_3$ (M=Al, Co, Fe, Gd) perovskites via chemical co-precipitation method

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Abstract

Nanocrystalline LaMO$_3$ (M = Al, Fe, Co, Gd) perovskites were synthesized by the co-precipitation method using metal nitrate and carbonate salts as starting materials. The products were characterized with X-ray diffractometer (XRD), energy dispersive X-ray spectrometer (EDX), scanning electron microscope (SEM), transmission electron microscope (TEM), Brunauer – Emmett – Teller (BET) specific surface area measurement, and UV-vis diffused reflectance spectroscopy (DRS). The XRD patterns confirmed the formation of perovskite phase. The SEM micrographs indicated that perovskite samples were nanosized particles with morphology containing porosity due to inter-particle voids. The measured pH$_{pzc}$ of LaAlO$_3$, LaCoO$_3$, LaFeO$_3$, and LaGdO$_3$ in this work were 8.6, 9.2, 9.0, and 8.1, respectively. From the DRS spectra, the band gap energies of LaAlO$_3$, LaCoO$_3$, LaFeO$_3$, and LaGdO$_3$ were calculated as 2.60, 1.50, 2.00, and 2.90 eV, respectively. The preparation of LaMO$_3$ (M = Al, Fe, Co, Gd) perovskites in this work have several advantages such as simplicity, low cost, and no waste compared with other methods.
Keywords: La-based perovskite, LaAlO$_3$, LaCoO$_3$, LaFeO$_3$, LaGdO$_3$, Chemical co-
precipitation method.

1. Introduction

Perovskite is mixed oxide of transition metals with chemical formula ABO$_3$
where A is transition metal or lanthanide series cation, B is transition metal cation, and
O is oxide anion (Farhadi et al., 2010). The structure of perovskite is a face-centered
cubic cell of cation A and anion O. In the unit cell, A locates at the cubic corner and B
at the center of the lattice (Athayde et al., 2016). Several properties such as structure,
electronics, and magnetic properties of these compounds depend on the type of cations
A and B. The property and use must be considered hand-in-hand, for example, the
perovskite for use as gas sensor should have A and B such that ABO$_3$ exhibits electronic
property as semiconductor or if the aim is in fuel cell the selected ABO$_3$ should have
high k dielectric property.

The La-based perovskite of LaMO$_3$ (M=Al, Co, Fe, Gd) are very interesting
compounds, they can find application in many fields. LaAlO$_3$ can be used as
thermoluminescent dosimeter (Hernandez et al., 2016) and photoluminescence material
(Fu et al., 2016; Alves et al., 2014) while LaCoO$_3$ find application in the field of
catalyst (Pang et al., 2016), fuel cell (Natite et al., 2016), and gas sensor (Shi et al.,
2015; Ding et al., 2015). The LaFeO$_3$ perovskite have been studied as photocatalyst
(Kumar et al., 2017), gas sensor (Cao et al., 2017; Chen et al., 2016; Wei et al., 2017),
and cathode for fuel cell (Choi et al., 2016). LaGdO$_3$ can be used as dielectric material
for pulses laser (Pavunny et al., 2011) and high k dielectric material in electronic device
(Pavunny et al., 2014). The efficiency of these materials depend on the synthesis
method (Athayde et al., 2016). Many methods are available for the synthesis of perovskite oxide in the group of La-based perovskite such as solid state reaction (Rodriguez et al., 2017), sol gel (Cao et al., 2017), solution combustion synthesis (Fu et al., 2016), electrospinning (Li et al., 2016), hydrothermal synthesis (Niu et al., 2016), co-precipitation method (Chandradass et al., 2014; Tompsett et al., 1998), EDTA-glycine process (Qin et al., 2013), and reverse microemulsion process (Tian et al., 2009), etc. These techniques, however, still have disadvantages such as involving several steps and sometimes requiring more than necessary starting reagents.

Our research group attempted to study the preparation of La-based oxide of nano-LaMO$_3$ perovskites via a simple co-precipitation method and investigated their inherited properties for further applications. In this study, 4 types of M (metal group 3A (Al as Al$^{3+}$), transition metal (Co as Co$^{2+}$ or Co$^{3+}$ and Fe as Fe$^{3+}$) and lanthanide metal (Gd as Gd$^{3+}$)) were covered. In 2014 and 2015 we reported preliminary results of syntheses and characterizations of LaAlO$_3$ and LaFeO$_3$ (Haron et al., 2014; Haron et al., 2015). In this article, more details of the said syntheses and characterizations including the extension to cover Co and Gd are reported. In all, this synthesis route has several advantages such as simplicity, low cost, and no waste compared with other routes that we deem it would be useful for others who just begin to work in this field.

2. Materials and Methods

2.1 Syntheses of LaMO$_3$ perovskites

LaMO$_3$ (M = Al, Co, Fe, Gd) perovskites were prepared with some modifications based on the co-precipitation method reported previously (Villoria et al., 2011) La(NO$_3$)$_3$·6H$_2$O and Al(NO$_3$)$_3$·6H$_2$O were used as starting materials for
preparation of LaAlO$_3$. A specified amount of each was dissolved in distilled water to make 1 M solutions and mixed together with vigorous stirring. Then aqueous solution of K$_2$CO$_3$·1.5H$_2$O (2 M) – same volume as one of nitrate solution was rapidly added. Small amount of NaOH solution (1M) was introduced to initiate precipitation. After the precipitation was complete, the precipitate was filtered and washed with distilled water several times until the washed water became neutral (pH =7). The product was then dried at 100 °C for 6 h to yield the “precursor” after which it was calcined at 700, 800, and 900 °C for 2 h to obtain the LaAlO$_3$ perovskite.

For LaCoO$_3$, LaFeO$_3$, and LaGdO$_3$ perovskites, the same procedure as above was applied but with Co(NO$_3$)$_2$·6H$_2$O, Fe(NO$_3$)$_3$·9H$_2$O, Gd(NO$_3$)$_3$·6H$_2$O as starting materials, respectively. Each precursor then was calcined at the planned temperatures for 2 h. For LaCoO$_3$ at 200, 400, 600 °C, LaFeO$_3$ at 600, 700, 800 °C, and LaGdO$_3$ at 1000, 1100, 1200 °C.

2.2 Characterization techniques

The crystalline phase of LaMO$_3$ (M=Al, Co, Fe, Gd) samples was investigated using the X-ray powder diffractometer, Philips PW 3710 (PHILIPS X’Pert MPD) employing Cu Kα ($\lambda = 0.154$ nm) radiation and a Ni filter, over the range 20 = 20 - 80 °. The average crystallite size (D) was calculated with the Debye-Scherrer equation, Eq. (1),

$$D = \frac{K\lambda}{B\cos\theta}$$

where D is the average crystallite size, K is a dimensionless shape factor with a typical value of about 0.89 but may vary with the actual shape of the crystallite. The $\lambda$ is the X-ray wavelength, B is the line width at half the maximum intensity (FWHM) after
subtracting the instrumental line broadening in radians, and $\theta$ is the Bragg angle. The metal constituents of LaMO$_3$ perovskite were investigated by using dispersive X-ray spectrometer (EDAX), ISIS 300, Oxford, England. The morphology of the LaMO$_3$ particles was examined using scanning electron microscope (SEM: JEOL, JSM – 5800 LV) operated at 20 kV in secondary electron imaging mode and transmission electron microscope (TEM: JEOL, JSM-2010) operated at 200 kV in bright-field mode. The specific surface area and pore size distribution were determined by nitrogen sorption isotherm using the BET method (Coulter, model SA 3100, USA).

2.3 Determination of pH at point of zero charge ($pH_{pzc}$)

A sodium chloride solution of 0.1 mol /L was prepared by dissolving an accurately weighed amount of NaCl salt in a 1 L volumetric flask. Several portions of 50 mL of this solution were pipetted and poured into several 250 mL Erlenmeyer flasks. From these solutions, the pH of 1 to 12 were made using diluted solutions of HCl or NaOH and recorded as initial pH, $pH_i$ (Liao et al., 1999)

About 0.05 g of LaMO$_3$ was added into each flask and sealed with constant agitation using a magnetic stirrer for 24 h. The stirring rate was controlled at 300 rpm. At the end of experiment, the equilibrated solution was decanted and the pH was measured as final pH, $pH_f$. A graph was constructed by plotting $pH_i$ against $pH_f$ and the $pH_{pzc}$ was determined from the crossover point of $pH_i$ and $pH_f$ in the graph.

2.4 The UV-vis diffused reflectance spectroscopy (DRS)

The UV-vis diffused reflectance spectra (DRS) were recorded from 200 nm to 800 nm. The band gap energy was then calculated using the Kubelka – Munk equation:
(Rhν)^2 = A (hv – Eg)  .................................. (2)

where R is the absorbance, hv is the absorption energy, A is the parameter related to the
effective masses associated with the valence and conduction bands, n is refraction
index, and Eg is the band gap energy (Stampler et al., 2008).

3. Results and Discussion

3.1 Syntheses of LaMO₃ products

For M = Al, Co, Fe, Gd: the colors of precursors obtained in the first stage
were white, violet, brown, and white, respectively. After calcination at 900 (M=Al), 600
(M=Co), 800 (M=Fe), and 1200 °C (M=Gd) for 2 h, the precursors turned to the
corresponding perovskites. The colors of perovskites were white, black, dark brown,
and white, respectively. The photographs showing color of precursors and perovskites
are displayed in Figure 1.

In this study, LaMO₃ (M = Al, Co, Fe, Gd) perovskite powders were prepared
by the co-precipitation method. The process may be described by the following
chemical reactions.

2 La(NO₃)₃ + 2 M(NO₃)₃ + 6 K₂CO₃ → La₂(CO₃)₃ + M₂(CO₃)₃ + 12
KNO₃

[Diagram]

La₂(CO₃)₃ + M₂(CO₃)₃ → La₂O₃ + M₂O₃ + 6CO₂

[Diagram]

La₂O₃ + M₂O₃ → 2 LaMO₃

where M = Al, Co, Fe, Gd; high temp 2 > high temp 1
The precursor first obtained was a mixture of $\text{La}_2(\text{CO}_3)_3$ and $\text{M}_2(\text{CO}_3)_3$ which later was subjected to calcination. As the temperature rose, the carbonates decomposed to the corresponding oxides and subsequently transformed to the corresponding perovskite at high temperature.

### 3.2 Products characterization

#### 3.2.1 X-ray diffractometer (XRD)

The formation of oxides prior to formation of perovskite phase was evidenced from the XRD patterns displayed in Figures 2-3. For $\text{LaAlO}_3$, perovskite phase was detected only at 900 °C as shown in Figure 2(a); at lower than 900 °C, the product was a mixture of $\text{La}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$. The XRD peaks at 900 °C matched with the hexagonal structures of $\text{LaAlO}_3$ perovskite (JCPDS Card No. 85-0848). The average crystallite size of this $\text{LaAlO}_3$ was 75 nm by using Eq. (1).

The XRD patterns of $\text{LaCoO}_3$ and $\text{LaFeO}_3$ obtained at various temperatures are shown in Figures 2(b) - 3(a). For $\text{LaCoO}_3$, the initial +2 charge was oxidized by oxygen during the heating stage to +3. The rhombohedral $\text{LaCoO}_3$ perovskite formed at 600 °C (matched with JCPDS file no. 48-0123), otherwise, the mixture of $\text{La}_2\text{O}_3$ and $\text{Co}_2\text{O}_3$ was obtained if the calcination temperature was lower than 600 °C. The crystallite size of $\text{LaCoO}_3$ at 600 °C was 69 nm. For $\text{LaFeO}_3$, the product obtained from calcination at 800 °C was $\text{LaFeO}_3$ perovskite (matched with JCPDS file no. 37-1493) having orthorhombic structure with crystallite size of 68 nm. If the calcination temperature was lower than 800 °C, only a mixture of $\text{La}_2\text{O}_3$ and $\text{Fe}_2\text{O}_3$ was obtained.

The perovskite phase of $\text{LaGdO}_3$ formed at very high temperature. From the XRD pattern, $\text{LaGdO}_3$, perovskite phase was detected only at 1200 °C, at lower
1200 °C, the product was mixture of La$_2$O$_3$ and Gd$_2$O$_3$. The XRD pattern of LaGdO$_3$ at 1200 °C matched with the JCPDS Card No. 42-1465 of monoclinic LaGdO$_3$ structure (Figure 3(b)). The XRD peak informations yielded the average crystallite sizes of 71 nm.

The appearances of XRD patterns of all four perovskites are sufficiently clean from other unwanted peaks such that they can be considered that these methods were able to produce the perovskite phase without waste compared with other methods.

The syntheses using the low temperature not only save energy but also increase surface area of perovskite products. The varying of calcination temperature for the synthesis LaFeO$_3$ was reported in 2015 from our group (Haron et al., 2015) while the same informations of LaAlO$_3$, LaCoO$_3$, and LaGdO$_3$ still have not appeared literatures. Hence, this research is the first to provide this information. The result of LaFeO$_3$ in this article was from the reinvestigation carried out simultaneously with the other three perovskites and still bears resemblance to that reported in 2015.

3.2.2 Energy dispersive x-ray spectrometer (EDX)

The purity of all samples were also checked with EDX spectrometer (Figure 4). From the EDX pattern, all the elements detected were those that were expected to be present in the samples, i.e. La, Al, Co, Fe, Gd, and O. In addition, all powder samples contain insignificant amounts of other contaminating elements, affirming high purity of synthesized perovskite oxides.

3.2.3 Scanning electron microscope (SEM)
The morphology of all samples were investigated by using scanning electron microscope (SEM). Figure 5 shows the SEM micrographs of the LaMO$_3$ samples at 10,000 and 30,000 magnifications, it can be clearly seen that each sample has its own characteristic morphology. The surface of LaAlO$_3$ appeared as mixing of 2 types of grains of cubic shape and nanorod-like shape. The cubics and short sticks aggregated to become clusters with some porosity. The nanorod was > 1µm in length with thickness of approximately 10 nm and the width of approximately 10-100 nm. These grain shapes made it to be more porous and should be beneficial to applications such as gas sensor and metal ions adsorbent (Figure 5(a)). In contrast, the SEM image of the LaCoO$_3$ perovskite revealed (Figure 5(b)) its the surface composed of small sized of grains with round shape and it can be seen that the surface of LaCoO$_3$ was very smooth. The LaFeO$_3$ perovskite (Figure 5(c)) exhibited large amount of round grains mixing with small amount of stick grains. The round grains of LaFeO$_3$ were small but they were still bigger than round grains of LaCoO$_3$. On the other hand, LaGdO$_3$ existed as long grains shape with very large size, the largest compared with LaAlO$_3$, LaCoO$_3$, and LaFeO$_3$ (length > 2 µm).

The surface morphologies of LaMO$_3$ (M = Al, Co, Fe, Gd) perovskites found in this work were both similar to and different from those previously reported by other groups. Li et al., 2007 studied the SEM of LaAlO$_3$ synthesized by molten salt route at 800 °C for 3 hrs, the surface of their sample had extended cubic shape particles similar to the surface of as-synthesized LaAlO$_3$ in this work. For LaCoO$_3$ prepared by other methods, the particle morphologies of LaCoO$_3$ were micron size with high degree of agglomeration composed of nanocrystallites with an average size of 11 nm (Ghasdi et al., 2010). Wang et al., 2013 studied the microstructure of LaFeO$_3$ powders prepared by
sol gel method and calcined at 800 °C for 2 h, the SEM of sample contained grains with large particles accompanied by smoother surfaces. In the case of LaGdO₃, no information has not been found in the literatures for comparison.

**3.2.4 Transmission electron microscope (TEM)**

From the TEM micrographs (Figure 6), LaAlO₃ appeared as porous material (Figure 6(a)) composed of nanorods containing internal pores throughout the structures. The diameter of pore were approximately 10-80 nm. This unique feature may provide additional beneficial from surface area for application to gas sensor, catalyst, and metal ions adsorption. LaCoO₃, also a porous material, had round grains (Figure 6(b)) while LaFeO₃ had stick-shape with round edges (Figure 6(c)). The last member of the group, LaGdO₃, exhibited very large grains, the largest of all four as-synthesized perovskites in this set (Figure 6(d)).

**3.2.5 Specific surface area measurement**

The BET surface area and pore volume of LaMO₃ at difference calcination temperatures based on the N₂ adsorption isotherms are given in Table 1. It can be seen that the nano LaAlO₃ has the highest surface area at 61.7 m²/g while both nano-LaCoO₃ and LaFeO₃ have almost identical surface area ~ 23 m²/g similar to those reported in the literatures. Both Co³⁺ and Fe³⁺ having equal ionic radii (=60 pm) could be the reason for equal surface area of LaCoO₃ and LaFeO₃. The surface area of LaGdO₃ is 5.4 m²/g, the lowest in the LaMO₃ group. This could be the result of large Gd³⁺ ionic size (93.5 pm) and its high atomic weight, hence, less unit cell per gram and low surface area per gram.
The surface area of synthesized LaAlO$_3$, LaCoO$_3$, and LaFeO$_3$ were not much different from those reported by other researchers as shown in Table 1. There has been no data reported for LaGdO$_3$.

3.3 pH at point of zero charge ($\text{pH}_{\text{pzc}}$)

The $\text{pH}_{\text{pzc}}$ determinations were carried out using the pH drift method (Liao et al., 1999). The equilibrated pH ($\text{pH}_e$) of the as-prepared LaMO$_3$ ($M = \text{Al, Co, Fe, Gd}$) were plotted against initial pH ($\text{pH}_i$) of the solution having constant ionic strength (0.1 mol/L NaCl). The pH at a point where the initial pH of the solution crossover the equilibrated pH is the $\text{pH}_{\text{pzc}}$ of the sample (Figure 7(a-c)). The measured $\text{pH}_{\text{pzc}}$ of LaAlO$_3$ (Figure 7(a)), LaCoO$_3$ (Figure 7(b)), LaFeO$_3$ (Figure 7(c)), and LaGdO$_3$ (Figure 7(d)) in this work were 8.6, 9.2, 9.0, and 8.1, respectively, indicating the surface of all synthesized LaMO$_3$ were slightly basic. The $\text{pH}_{\text{pzc}}$ of LaAlO$_3$, LaCoO$_3$ in this work were not much different from those reported at 9.9 (Negahdari et al., 2009) and 12.8 (Mezaini et al., 2014), respectively, while there were no data for LaFeO$_3$ and LaGdO$_3$.

3.4 The UV-vis diffused reflectance spectroscopy (DRS)

For the DRS spectra of LaMO$_3$ ($M = \text{Al, Co, Fe, Gd}$) in the range 200 – 800 nm, the details of spectra were observable only in the range 200 – 400 nm for all samples. The band gap energy was calculated using the Kubelka – Munk (Figure 8(a-c)) and the obtained band gap energies of LaAlO$_3$, LaCoO$_3$, and LaFeO$_3$ were 2.60, 1.50, 2.00, and 2.90 eV respectively. This result confirmed that all LaMO$_3$ perovskites were all semiconductor materials.
Materials for application as gas sensor and heavy metal adsorbent should be nanomaterial, porous material, high surface area, and semiconductor (for gas sensor). LaAlO$_3$ from this work is nanocrystalline perovskite with the highest surface area among all perovskites synthesized as well as being a semiconductor material. These properties put LaAlO$_3$ as the most promising candidate for further investigation in the area of gas sensor and metal ions adsorption for wastewater treatment.

4. Conclusion

Nanocrystalline LaMO$_3$ (M = Al, Co, Fe, Gd) perovskites in powder form were prepared using the co-precipitation method at lower calcination temperatures, i.e. lower than conventional methods. The products were characterized with XRD, EDX, SEM, TEM, and BET which revealed significant differences in physical properties of the samples. We found that LaAlO$_3$ showed very interesting properties among all four perovskites prepared such as high surface area, and semiconductor property with band gap =2.60 eV. We hope that LaAlO$_3$ synthesized from this work will be standout in the applications such as gas sensing material and heavy metal ions adsorbent. The syntheses of LaMO$_3$ (M = Al, Co, Fe, Gd) perovskites in this work have several advantages such as simplicity, low cost, and no waste compared with other methods. Parts of the data appear in this report are the first ever to be reported.

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References


New Electronic Device Material for CMOS Logic and Memory Devices.

Materials. 7, 2669-2696.


Figure 1. The precursors prepared by the chemical co-precipitation method and perovskite oxides after being calcined at 900 °C (LaAlO$_3$), 600 °C (LaCoO$_3$), 800 °C (LaFeO$_3$), and 1200 °C (LaGdO$_3$) for 2 h: (a1) LaAlO$_3$ precursor, (a2) LaAlO$_3$ perovskite, (b1) LaCoO$_3$ precursor, (b2) LaCoO$_3$ perovskite, (c1) LaFeO$_3$ precursor, (c2) LaFeO$_3$ perovskite, (d1) LaGdO$_3$ precursor, and (d2) LaGdO$_3$ perovskite.
Figure 2. XRD patterns of products from (a) LaAlO$_3$ and (b) LaCoO$_3$ obtained from various calcination temperatures for 2 h.
Figure 3. XRD patterns of (a) LaFeO$_3$ and (b) LaGdO$_3$ obtained from various calcination temperatures for 2 h.
Figure 4. EDX spectra of (a) LaAlO$_3$, (b) LaCoO$_3$, (c) LaFeO$_3$, and (d) LaGdO$_3$ powders prepared by co-precipitation after calcination at 900, 600, 800, and 1200 °C.
Figure 5. SEM images at x10,000 and x30,000 magnifications of (a) LaAlO$_3$, (b) LaCoO$_3$, (c) LaFeO$_3$, and (d) LaGdO$_3$ powders prepared by co-precipitation method.
Figure 6. TEM images of (a) LaAlO$_3$, (b) LaCoO$_3$, (c) LaFeO$_3$, and (d) LaGdO$_3$ powders prepared by co-precipitation.
Figure 7. Plot for the determination of $pH_{pzc}$ of (a) LaAlO$_3$, (b) LaCoO$_3$, (c) LaFeO$_3$, and (d) LaGdO$_3$. 
Figure 8. Band gap energy determination using Kubelka – Munk plot for (a) LaAlO$_3$, (b) LaCoO$_3$, (c) LaFeO$_3$, and (d) LaGdO$_3$. 

band gap = 2.60 eV

band gap = 1.50 eV

band gap = 2.00 eV

band gap = 2.90 eV
Table 1. BET surface area and total pore volume of LaMO$_3$ (M=Al, Co, Fe, Gd) prepared by co-precipitation method.

<table>
<thead>
<tr>
<th>Perovskite LaMO$_3$</th>
<th>Calcine temp. (°C)</th>
<th>Total pore volume (cc/g)</th>
<th>Surface area (m$^2$/g)</th>
<th>Surface area reported in literature (m$^2$/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LaAlO$_3$</td>
<td>900</td>
<td>0.13</td>
<td>61.7</td>
<td>43.0 (Negahdari et al., 2009)</td>
</tr>
<tr>
<td>LaCoO$_3$</td>
<td>600</td>
<td>0.06</td>
<td>23.1</td>
<td>27.0 (Farhadi et al., 2010)</td>
</tr>
<tr>
<td>LaFeO$_3$</td>
<td>800</td>
<td>0.07</td>
<td>23.0</td>
<td>36.5 (Farhadi et al., 2009)</td>
</tr>
<tr>
<td>LaGdO$_3$</td>
<td>1200</td>
<td>0.03</td>
<td>5.4</td>
<td>No data</td>
</tr>
</tbody>
</table>