

Synthesis and Characterization of Exfoliated Layered $K_2La_2Ti_3O_{10}$ Perovskite Oxide

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Photocatalytic water splitting is one of the renewable energy techniques in an eco-friendly manner, which plays an essential role in addressing the challenges associated with conventional fossil fuels [1–3]. Photocatalytic water splitting is a production of hydrogen via direct conversion of solar energy to chemical energy, as discovered by Fujishima and Honda in 1972 using TiO_2 as photoelectrode [4]. In the past few years, researchers focused on developing efficient photocatalysts to produce hydrogen gas in cost-effective ways to reach industrial scale. Several catalysts have found to be effective; and among them, layered perovskite oxides displayed significant photocatalytic properties. Layered perovskites are derivatives from the simple perovskites ABO_3 , where A is an alkaline or alkaline earth or rare earth element, and B is a transition element [5]. Layered perovskites consist of slabs of ABO_3 and they are classified based on orientation of layer stacking. The layered perovskites showing orientation along (111) and (110) can be expressed with a formula of $A_{n+1}B_nO_{3n+3}$ and $A_nB_nO_{3n+2}$, respectively; and along (100) plane, expressed as $(Bi_2O_2)(A_{n-1}B_nO_{3n+1})$ (Aurivillius phase, AL), $A_{n+1}B_nO_{3n+1}$ (Ruddlesden–Popper phase, RP) and $A'(A_{n-1}B_nO_{3n+1})$ (Dion–Jacobson phase, DJ), where n represents the number of BO_6 octahedra arranged perpendicular to the layers [6,7].

Bulk layered perovskites have exhibited good photocatalytic activity if they are modified using ion-doping, when combined with cocatalysts. Nanosheets of layered perovskites possess high catalytic surface areas, which is advantageous for charge transfer process [8]. Hence, by exfoliation of bulk layered perovskite, it is an efficient way of designing promising catalysts using the nanosheets constructing efficient photocatalysts via restacking.

In this work, we synthesized $K_2La_2Ti_3O_{10}$ (KLTO) DJ layered perovskite, which was modified by exfoliation to enhance the photocatalytic properties. Initially, bulk $K_2La_2Ti_3O_{10}$ is prepared using modified Pechini method by taking stoichiometric ratios of potassium and lanthanum nitrate salts and TiO_2 by using citric acid and ethylenediaminetetraacetic acid as a chelating agent at pH 9. The reaction mixture was first carbonized at 250 °C, and later calcined at 1,000 °C. The bulk KLTO was exfoliated utilizing *n*-butyl lithium (*n*-BuLi) as exfoliating agent. 3 mol excess of *n*-BuLi was added to the powder of bulk KLTO perovskite powder in a hexane and stirred continuously for three days, and the lithiated KLTO was obtained. The as-obtained lithiated KLTO was washed with hexane and dried in vacuum at room temperature. The exfoliated KLTO suspended in distilled water and centrifuged at 10,000 rpm, and washed thoroughly with distilled water to remove LiOH by confirming the pH of supernatant 7. The washed product was redispersed in distilled water and ultra-sonicated for 60 min. SEM observation confirms that the bulk KLTO is irregular particles in micron meter size (Figure 1). Figure 2a and b depict the low and high magnification SEM images of exfoliated KLTO, which shows successful exfoliation and formation nanometer size sheets. This method offers a facile way of producing exfoliated nanosheets of layered perovskites, which can be extended to other perovskite-based oxides for efficient design of cost-effective photocatalysts for water splitting [9].

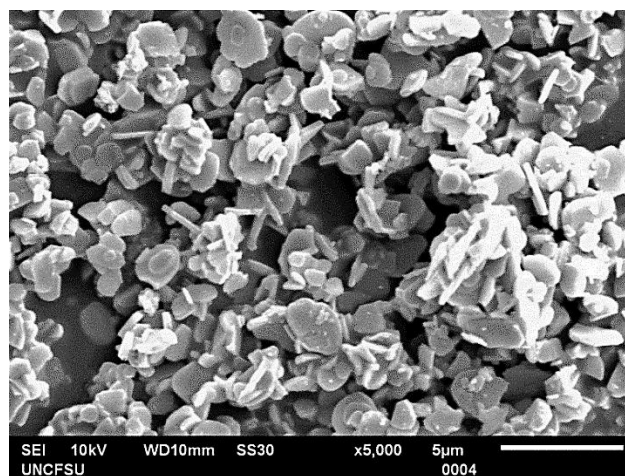


Figure 1. SEM image of KLTO layered perovskite oxide powders.

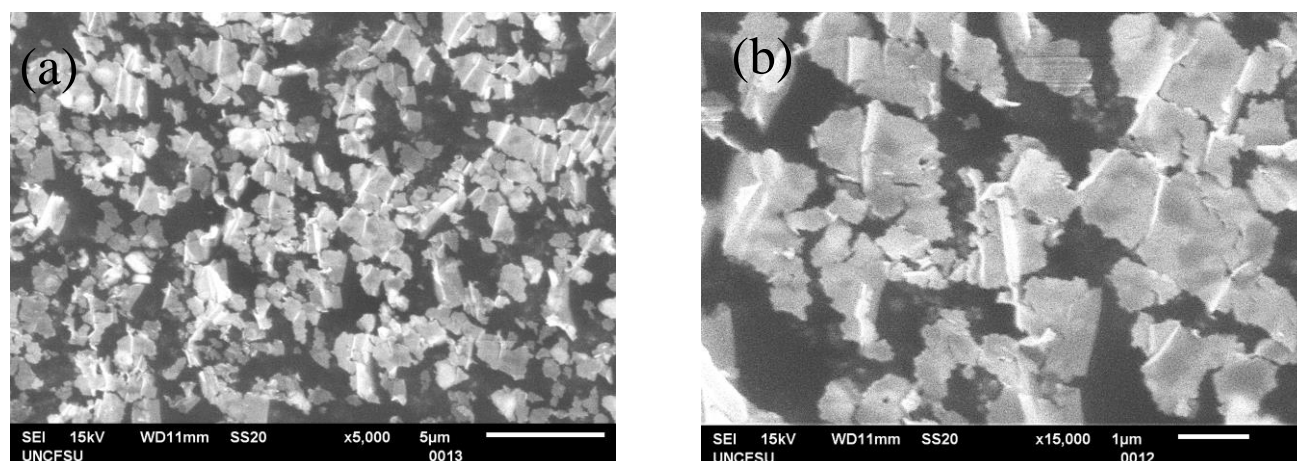


Figure 2. SEM images of exfoliated KLTO perovskite oxide powders at a low magnification (a) and high magnification (b).

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