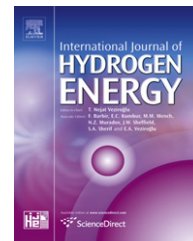


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# Photocatalytic water splitting on new layered perovskite $A_{2.33}Sr_{0.67}Nb_5O_{14.335}$ ( $A = K, H$ )

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## ABSTRACT

A new layered compound  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was prepared and its full structure was characterized by single crystal diffraction of  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$ , which belongs to Tetragonal layered perovskite phase. The band structure of  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was calculated by CASTEP code based on the density functional theory (DFT).  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was prepared by a proton exchange of  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$ , and followed with successive reaction to obtain Pt incorporated sample. Using  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}/Pt$  as catalyst, the photocatalytic  $H_2$  evolution rate reached  $153.1 \text{ cm}^3 \text{ h}^{-1} \text{ g}^{-1}$  in 10 vol.% methanol aqueous solution under irradiation with wavelength more than 290 nm from a 100 W mercury lamp.

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## 1. Introduction

Hydrogen is considered as an ideal fuel for the future. However, renewable energy contributes only about 5% of the commercial hydrogen production primarily via water electrolysis, while other 95% hydrogen is mainly derived from fossil fuels presently. Renewable hydrogen production is not popular yet because the cost is still high. Photovoltaic water electrolysis may become more competitive as the cost continues to decrease with the technology advancement. Photocatalytic water splitting using semiconductor for hydrogen production offers a promising way for clean, low-cost and environmentally friendly production of hydrogen by solar energy [1–5]. Since the first photocatalyst titanium dioxide suitable for hydrogen evolution from water splitting was reported [1] several decades ago, considerable efforts [2–8] have been devoted to develop a semiconductor photocatalyst for practical application.

Ion-exchangeable layered perovskites compound composed of alternative stacking of a two-dimensional perovskite slab with different numbers of layers and monovalent cations. Owing to the layered structure, water molecules and other medium can easily enter the interlayer and react with the layered compound. On the other hand, the layered compound can use their abundant interlayer space as reactions sites, and the recombination of the electrons and holes generated by photoinduction could be restrained. Beside, due to layered structure, various substances, such as promoters, can be intercalated into the interlayer to improve the photocatalytic capability of the compound [9–19]. In this paper, a new layered perovskite compound  $A_{2.33}Sr_{0.67}Nb_5O_{14.335}$  ( $A = K, H$ ) is prepared by a conventional solid-state reaction and proton exchange reaction. The structure, band energy and photocatalytic properties are investigated. It is found that the compound  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}/Pt$  shows good photocatalytic activities for water decomposition.

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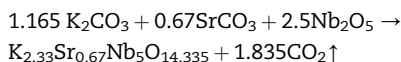
**Table 1 – Data collection.**

Diffractometer used	Rigaku AFC7R
Radiation source	Fine-focus sealed tube
Radiation type	MoK $\alpha$ 0.71073A
Temperature(K)	293(2)
Monochromator	Graphite-monochromator
Theta range (deg)	2.31–25.49
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50.0% of total scan time
Standard reflections	3 Measured every 200 reflections
Reflections measured	681
Index ranges of measured data	$-4 \leq h \leq 15, 0 \leq k \leq 15, -4 \leq l \leq 1$
Independent reflections	347 (Rint = 0.0752)
Observed reflection	312 ( $>2\sigma(I)$ )
Empirical absorption correction	Psi (Rigaku WinAFC)
Relative transmission factor	0.2692–0.9681

## 2. Experimental

### 2.1. Sample preparation

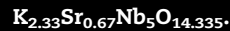
The layered compound  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was prepared by a conventional solid-state method. The stoichiometric mixture of carbonates ( $K_2CO_3$ ,  $SrCO_3$ ) and oxides ( $Nb_2O_5$ ) according to the following reaction was mixed and grinded for 1 h. An excess amount of alkali metal carbonate  $K_2CO_3$  (10 mol.%) was added to compensate for the loss due to the volatilization of alkali component during the calcination. Then the mixture was calcined at 1600 °C for 1 h in air. After cooling to room temperature, the compound  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was obtained.



$H_{2.33}Sr_{0.67}Nb_5O_{14.335}$  was prepared by the proton exchange reaction of  $K_{2.33}Sr_{0.67}Nb_5O_{14.335}$  in 1 M HCl solution at 40 °C for 4 days with intermediate replacement of the acid each 24 h.

**Table 2 – Solution and refinement.**

System used	Siemens SHELXTL PLUS (PC Version)
Structure solution	Siemens SHELXTL
Structure refinement	Siemens SHELXTL
Refinement method	Full-matrix least-squares on $F^2$
Extinction coefficient	0.022(3)
Weighting scheme	calc $w = 1/[\sigma^2(F_o^2) + (0.0800P)^2 + 9.1220P]$ where $P = (F_o^2 + 2F_c^2)/3$
Parameter/restraints/data(obs.)	46/2/312
Final R indices (obs.)	$R1 = 0.0445, wR2 = 0.1160$
R indices (all)	$R1 = 0.0507, wR2 = 0.1213$
Goodness-of-fit	1.032
Largest and Mean Delta/Sigma	1.456, 0.032
Largest difference peak(e.A $^{-3}$ )	1.698, -2.039

**Table 3 – Structure determination summary for**

Crystal data	
Empirical formula	K Nb O Sr
Color and habit	Colorless prismatic
Crystal size (mm)	0.20 0.15 0.10
Crystal system	Tetragonal
Space group	P4/mbm
Unit cell dimensions	
a (Å)	12.449(3)
b (Å)	12.449(3)
c (Å)	3.8961(6)
Alpha (deg.)	90
Beta (deg.)	90
Gamma (deg.)	90
Volume(Å $^3$ )	603.8(2)
Z	6
Formula weight	235.63
Density(cal) (Mg/m $^3$ )	3.888
Absorption coefficient (mm $^{-1}$ )	16.904
F(000)	636

After the reaction, the solid product was washed with distilled water to remove the excess of the remained acid and then air-dried.  $[Pt(NH_3)_4]^{2+}$  was incorporated in the interlayer of  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}$  by stirring  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}$  (4 g) in 0.6 mM  $[Pt(NH_3)_4]Cl_2$  aqueous solution (1000 ml) at room temperature for 72 h. After being filtered and washed with water, the specimen was dispersed in water and irradiated with UV light from a 450 W high-pressure mercury lamp at room temperature for 5 h in order to deposit Pt particles in the interlayer of  $H_{2.33}Sr_{0.67}Nb_5O_{14.335}$ .

### 2.2. Sample characterization

Data collection: CrystalClear (Rigaku, AFC7R). Cell refinement: CrystalClear. Data reduction: CrystalClear. Program(s) used to solve structure: SHELXL97 (Sheldrick, 2008). Program(s) used to refine structure: SHELXL97 (Sheldrick, 2008). Molecular graphics: SHELXTL (Sheldrick, 2008). Software used to prepare material for publication: SHELXTL. The crystal structure of powdery sample was confirmed by using of a powder X-ray diffractometer (XRD, Bruker D8 Advance) with

**Table 4 – Atomic coordinates and equivalent isotropic displacement parameters (Å $^2$ ).**

Atom	x	y	z	U(eq)	SOF
Nb1	0.07554(8)	0.21202(8)	0.0000	0.0047(6)	1
Nb2	0.5000	0.0000	-1.0000	0.0106(7)	1
Sr1	0.0000	0.0000	-0.5000	0.0054(14)	0.22(2)
K1	0.0000	0.0000	-0.5000	0.0054(14)	0.78(3)
Sr2	0.3289(2)	0.1711(2)	-0.5000	0.037(2)	0.23(2)
K2	0.3289(2)	0.1711(2)	-0.5000	0.037(2)	0.77(3)
O1	0.2217(11)	0.2783(11)	0.0000	0.046(5)	1
O2	0.1387(12)	0.0664(11)	0.0000	0.064(5)	1
O3	-0.0064(13)	0.3437(10)	0.0000	0.075(6)	1
O4	0.5000	0.0000	-1.5000	0.11(2)	0.84
O5	0.076(2)	0.2057(17)	-0.5000	0.097(11)	0.87

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