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LUMINESCENT PROPERTIES OF PRASEODYMIUM-DOPED PHOSPHATE TUNGSTEN BRONZE

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ABSTRACT:

Nowadays phosphate tungsten bronzes (PWBs) attract a lot of attention due to their interesting chemical, mechanical, and optical features. Moreover, tungsten bronzes as inert inorganic solids, with incorporated rare-earth ions in their structure, show interesting and useful electronic properties. Praseodymium doped phosphate tungsten bronze (Pr-PWB) is obtained in the process of phase transformations of $PrPW_{12}O_{40}\cdot 6H_2O$ (Pr-PWA) salt. The green crystals of Pr-PWB are formed after the heating of Pr-PWA in a furnace, in a temperature range from room temperature to 650 °C. In the present paper the fluorescent properties are analyzed of Pr-PWB, its precursor – 12-tungstophosphoric heteropoly acid, $H_3PW_{12}O_{40}\cdot 29H_2O$ (PWA) with Keggin's anion structure, as well as the intermediate – Pr-PWA salt. The luminescent properties where characterized and the obtained results showed that both samples emit in the deep blue region, indicating their potential use as a blue emitting source for white light LED's.

Keywords: *phosphate tungsten bronzes, praseodymium, luminescent properties*

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1. INTRODUCTION

Keggin-type heteropolyacids (HPAs) with the general formula $H_{(8-x)}XM_{12}O_{40}\cdot nH_2O$ (X^{x+}=P^{5+}, Si^{4+}, As^{5+}, Ge^{4+}, Ce^{4+}, Th^{4+}, where x is the oxidation number of X, M=Mo, W, V, Nb and n=6-31), have been of interest in basic and applied science for more than a century because of their high protonic conductivity at room temperature [1-3]. As shown by our previously conducted studies of 12-tungstophosphoric praseodymium salt 6-

hydrate at high temperature, this heteropoly compound could be used as a precursor for the synthesis of praseodymium doped phosphate tungsten bronze (Pr-PWB) [4-7]. Phosphate tungsten bronzes (PWBs) continue to attract considerable attention, due to their interesting chemical, mechanical, electrical, and optical properties. As inert inorganic solids with alkali, alkaline earth, or rare earth ions incorporated into their structure, PWBs exhibit particularly interesting and useful electronic and magnetic features [8]. The structure of PWBs can be described as a ReO_3-like structure, divided by slices of phosphate (PO_4) or diphosphate (P_2O_7) groups and a collection of WO_6 units repeated along three bearings by common corners [9-11]. This type of structure allows the formation of cavities into which various ions can be inserted. The incorporation of small cations such as Li^+ and Na^+ leads to the formation of perovskite-type and tetragonal-type PWBs, while the incorporation of larger cations such as K^+, Rb^+, Cs^+ leads to the formation of hexagonal-type PWBs [11]. A further subdivision of PWBs can be made by substituting WO_6 units into ReO_3 structures: monophosphate tungsten bronzes with pentagonal channels, in which one WO_6 octahedron is replaced by a PO_4 tetrahedron, and diphosphate tungsten bronzes with hexagonal channels, in which two adjacent WO_6 octahedra are replaced by a P_2O_7 group consisting of two PO_4 tetrahedra with a common O atom [9]. This work deals with monophosphate tungsten bronze doped with praseodymium ions, which belong to the rare earth group. In this work, the luminescence properties of praseodymium doped PWB (Pr-PWB), its precursor-12-tungstophosphoric heteropoly acid, $H_3PW_{12}O_{40}\cdot 29H_2O$ (29-PWA), and the intermediate - Pr-6-PWA salt, are analyzed to obtain more information about their behavior and possible practical applications.

2. EXPERIMENTAL

2.1. Synthesis of HPA hydrate $H_3PW_{12}O_{40}\cdot 6H_2O$ (6-PWA)

The HPA 29-hydrate solution (29-PWA) was prepared by mixing the aqueous $Na_2WO_4\cdot 2H_2O$ solution, prepared by dissolving 100 g $Na_2WO_4\cdot 2H_2O$ (Carl Roth, Germany) in 100 mL distilled water with the H_3PO_4-HCl mixture, prepared by mixing 10 mL 85% H_3PO_4 (Merck, Germany) with 80 mL 35% HCl (Merck, Germany). The precipitate was then extracted with 40 mL of 35% HCl and 70 mL of ether (Merck, Germany) at room temperature [12]. HPA 6-hydrate was obtained by the dehydration method by heating 29-PWA in an oven at 80 °C [4]. Subsequently, 6-PWA was used as a starting material for the synthesis of doped PWB.

2.2. Synthesis of Pr-PWA and Pr-PWB

Pr-PWA was prepared by mixing the aqueous solution of 6-PWA, prepared by dissolving 8 g of 6-PWA in distilled water, with an aqueous $PrCl_3\cdot H_2O$ solution, prepared by dissolving 0.7102 g of $PrCl_3\cdot H_2O$ (Acros Organics, Belgium) in distilled water. The obtained solution was slightly heated and left overnight at room temperature to complete the crystallization process. Subsequently, the synthesized Pr-PWA salt was heated in a

furnace in a temperature range from room temperature to 650 °C (at 10 °C min^{-1}), and greenish crystals of Pr-PWB were formed.

2.3. Luminescence measurements

Luminescence measurements were performed using an Edinburgh Instruments FLSP920 UV-Vis-NIR spectrometer. A 450W Xe lamp was used as a stationary excitation source. Time-resolved measurements were recorded using a 60 W Xe lamp at a frequency of 100 Hz. A Hamamatsu R928P photomultiplier tube was used to record the emission signal in the visible region. All luminescence measurements were recorded at room temperature. Powders were sandwiched between quartz plates (Starna cells for powder samples, type 20/C/Q/0.2). The time-resolved measurements were fitted with a monoexponential function.

3. RESULTS AND DISCUSSION

The photoluminescence properties of PWA, PWB, Pr-PWA and Pr-PWB have shown only the phosphorescence of the matrix PWA and PWB, while the dopant Pr^{3+} has no significant effect on the change of emission spectra with characteristic emission for this ion. When excited in one of the W=O charge transfer bands, the emission of Pr^{3+} was not observed, which can be attributed to a low doping fraction of this lanthanide ion or to its inability to integrate into the PWA and PWB matrix, making it impossible to excite the lanthanide ion. These results may be due to the different synthetic route of the PWA precursors and PWB, which are synthesized in a slightly different way compared to the Keggin-type single-crystalline polyoxometallates (POMs), which may affect the polycrystalline structure in which the lanthanide ions may be coordinated differently while shifting the charge transfer band. The matrix itself exhibits fluorescence properties with broadband emission in the range of 400 to 500 nm. The matrix doped with Pr^{3+} does not show the characteristic emission peaks of the Pr^{3+} ion emission spectrum. The decay dynamics of the matrices and the matrices doped with praseodymium ions showed no differences, suggesting that there is no energy transfer from the matrix to the Pr^{3+} ions. The results of the lifetime measurements of pure matrices and doped samples are shown in Table 1 and Fig. 1.

Table 1. The results of lifetime measurements of pure matrixes and doped samples

Sample	τ_1 *(μs)*	R^2
PWA	2.91	0.9938
Pr-PWA	3.29	0.9834
PWB	2.66	0.9860
Pr-PWB	2.70	0.9842

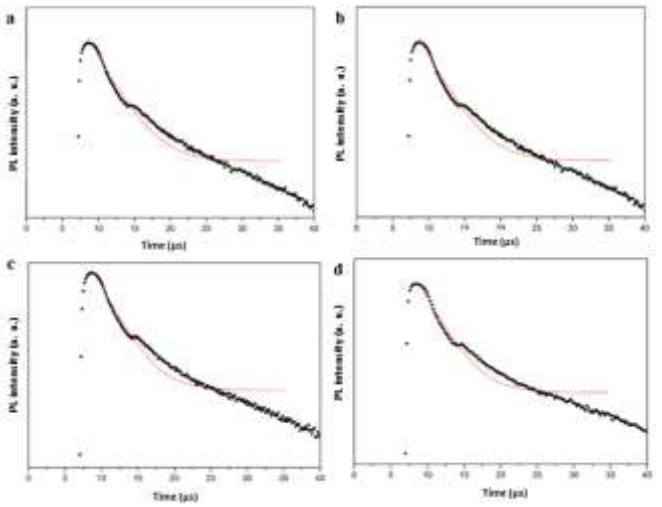

Fig. 1. Decay profiles of: **a** PWA excited at 320 nm, **b** Pr-PWA, **c** PWB, and **d** Pr-PWB excited at 376 nm and measured at room temperature

The CIE chromaticity diagrams have shown that all samples emit in deep blue region which can exhibit potential use as blue emitting source for white light LEDs (presented in Fig. 2).

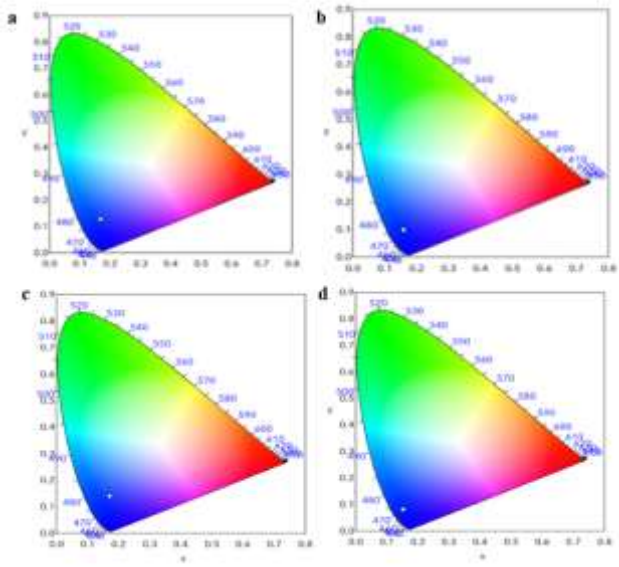

Fig. 2. The CIE chromaticity diagrams of: **a** PWA (x=0.167, y=0.127), **b** Pr-PWA (x=0.158, y=0.099), **c** PWB, and **d** Pr-PWB excited at 376 nm

3. CONCLUSION

In the present work, Pr-PWB is obtained by thermal conversion of Pr-PWA starting from 12-tungstophosphoric heteropolyacid as precursor. Praseodymium, a lanthanide ion belonging to the group of rare earth metals, was used for the first time, as a dopant for PWB. The green crystals of Pr-PWB form after heating Pr-PWA in a furnace, in a temperature range from room temperature to 650 °C. In this work, the luminescence properties of all undoped and doped samples were analyzed: PWA, Pr-PWA, PWB and Pr-PWA. The results obtained show that all samples emit in the deep blue range, which may represent a potential use as a source of blue emission for white light LEDs.

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