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Characterization of KNb_3O_8 Powders by Raman Spectroscopy and Scanning Electron Microscopy

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Potassium Triniobate KNb_3O_8 is an inorganic material that has promising photocatalytic applications for removing pollutants from wastewater [1]. Although there are different approaches to obtain this material, solid-state reactions at high temperature are used for obtaining powders of KNb_3O_8 [2]. Herein, we report the synthesis of KNb_3O_8 powders by solid-state reaction at 800˚C for 3h of Nb_2O_5 and KCl.

0.26581 g (1 mmol) of Nb_2O_5 (99.5%, Alfa Aesar, USA), 0.3727 g (5 mmol) of KCl (99.0 % Sigma-Aldrich, México) and 100 µL of deionised water were mixed to form. The paste was ground in an agate mortar for 20 min. Then, the powders were placed in a conventional furnace and heated at 800˚C for 3h. The powders were structurally characterised through Raman spectroscopy using a HORIBA Scientific LabRAM HR spectrometer integrated with laser light of 632 nm and by Field Emission Scanning Electron Microscopy (FESEM) within the JEOL JSM-7800F microscope.

Figure 1 shows the Raman spectrum of the powders. The peak maxima correspond to the vibrational modes of the KNb_3O_8 phase and are in good agreement with the literature [3]. This characteristic confirmed that after the solid-state reaction at 800°C for three hours, the KNb_3O_8 phase was obtained.

The elemental X-ray mapping in Figure 2 shows a homogeneous composition of the KNb_3O_8 compound obtained after calcination. The SEC image shows the microstructure of the sample, which consists of particles with different morphologies; flake-like and needle-like. The flake-like particles were coarser than the needle-like particles which seem to be formed as a result of breakage of the flake-like particles. The needles are approximately 150 nm x 1-3 µm. The thickness of the flakes is between 50-100 nm.

The method described here produced KNb_3O_8 powders at 800°C for 3 hours. Apparently, the grinding process allowed a homogeneous distribution of the elements, which favours the diffusion of K into the lattice of Nb_2O_5 to yield KNb_3O_8. This result opens the possibility to explore the synthesis of other systems through this methodology [4].

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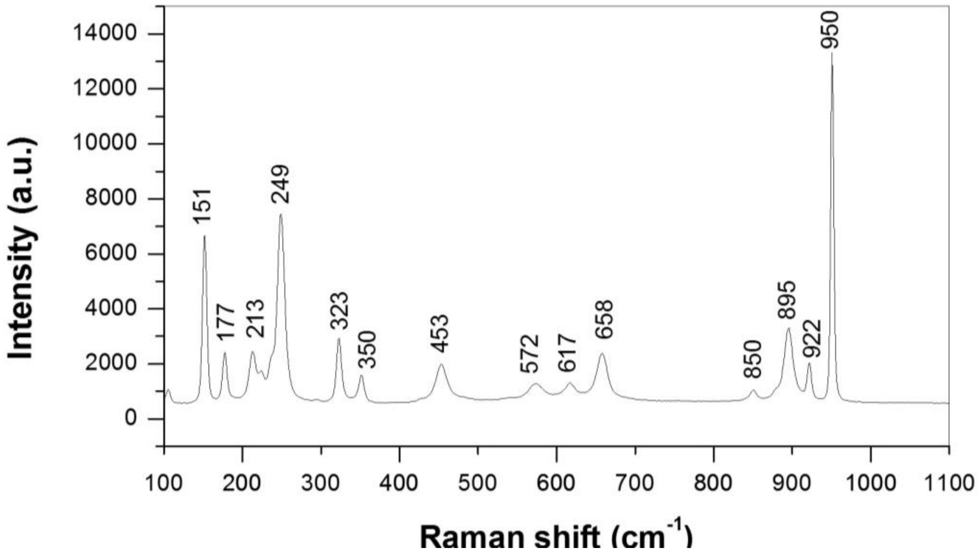

Figure 1. Raman spectrum of the powders of KNb3O8 obtained by calcination at 800°C for 3 hours.

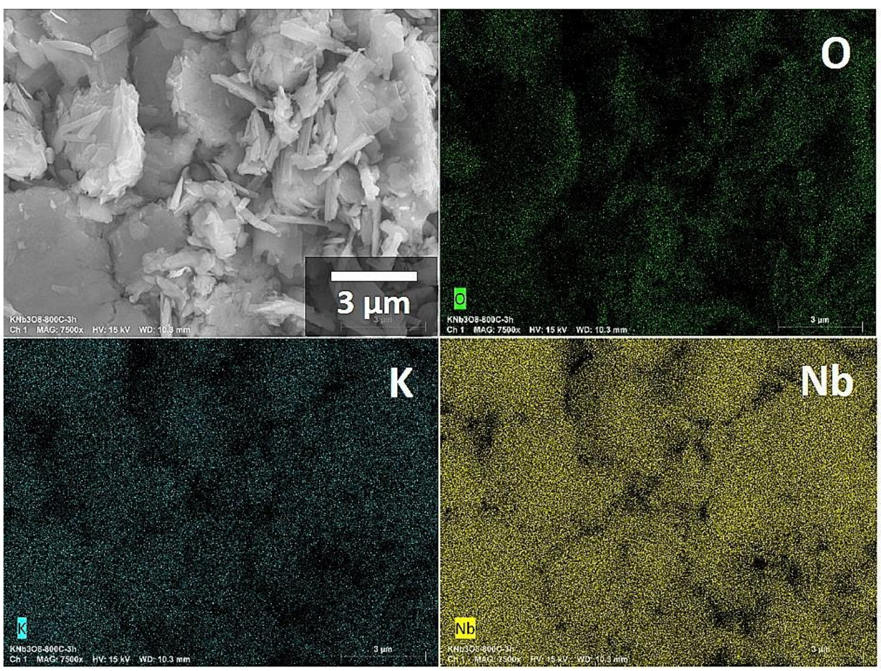

Figure 2. SEM image and elemental mapping of the powders of KNb3O8 obtained by calcination at 800°C for 3 hours.

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