Mixed CeO$_2$-Nb$_2$O$_5$-Bi$_2$O$_3$ nanoparticles were prepared using a resin-gel synthesis [1] and subsequently analysed using transmission electron microscopy (TEM), energy dispersive X-ray analysis (EDS), powder X-ray diffraction (PXRD), and X-ray photoelectron spectroscopy (XPS). These oxides are of importance because of their interesting crystal structures and industrial applications [2-4]. CeO$_2$ and δ-Bi$_2$O$_3$ are both excellent ionic conductors, making them suitable as the electrolyte component in solid oxide fuel cells (SOFCs). Nb$_2$O$_5$ has been used mainly as a solid support to take part in redox reactions and as a solid acid catalyst. As each of the parent metal oxides possesses useful properties, if all three metal ions can be contained in one particle, they could show novel structures and characteristics.

The primary purpose was to form nanoparticles containing appreciable proportions all three metal atoms, as at present there have been no investigations into the quaternary system CeO$_2$-Nb$_2$O$_5$-Bi$_2$O$_3$. The nanoparticles were then probed to see what crystal structures and atomic arrangements were exhibited, as it was assumed that, due to the more relaxed crystal structure prevailing at the nanoscale as compared to the bulk material, alternative cations would be more easily accommodated, hence facilitating the formation of a solid solution.

The mixed metal oxide nanoparticles were synthesized via a resin-gel method using polyethylene glycol (PEG mw=20,000) as the binding agent. The method used low temperatures (350 °C) to calcine the samples and prevent sintering of the nanoparticles. The synthesis was successful in producing mixed metal oxide nanoparticles with Scherrer analysis indicating crystallite sizes of 5-8 nm. EDS analysis showed many of the crystalline nanoparticles contained all three metals, however, elemental compositions calculated from EDS data varied significantly with elemental compositions calculated from XPS data. This evidence suggests that the distribution of elements within the particles is not homogenous, with bismuth showing a strong preference for surface or near-surface sites. Using FFT data from the electron microscope, d-spacings for the crystal lattices could be calculated. These values corresponded to the fluorite, pyrochlore and perovskite phases. This agrees with the PXRD data, which shows peaks indicative of these three phases.


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Fig. 1: (a) HRTEM micrograph with showing a highly crystalline region of the CeNbBi oxide with d-spacings calculated from the FFT giving values of 6.16 Å, 3.21 Å, and 2.83 Å indicating the pyrochlore structure has been formed. The d-spacings correspond approximately to the {111}, {311} and {400} lattice planes respectively.

Fig. 2: (b) FFT of the region of the CeNbBi oxide shown in (a).

Fig. 3: (c) EDS spectrum of the area imaged in (a).