Influence of heat treatment on the production of mixed oxides of Nb and Ta from columbite

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In this work a heat treatment study in a mixture of niobium and tantalum oxides extracted from the columbite mineral was carried out. Initially the mineral was physically prepared, followed by XRD and XRF characterizations. Subsequently, the mineral was leached with hydrochloric acid to remove iron and manganese present in its composition. Further leaching was performed with a mixture of hydrofluoric acid and sulfuric acid in order to solubilize the oxides of Nb and Ta and thus separate them from the persistent cassiterite impurity in the mineral. The hydrated oxides were precipitated with NH4OH and characterized by XRF, XRD and SEM. Then, the oxides were calcined at 800 °C, 900 °C and 1000 °C with varying heating rates and calcination times to follow the crystallization process of the oxides. The mixed oxides of Nb and Ta were characterized by XRF, XRD, SEM and physical adsorption of N2. The influence of the heating rate was verified in the tests at 800 °C and an increase in crystal size was observed when the rate was reduced from 10 ° C.min-1 to 5 ° C.min-1. Greater crystallinity and peak formation relative to the change from the hexagonal phase to the orthorhombic phase of the oxides was observed as the calcination temperature was raised from 800 °C to 900 °C. At the temperature of 1000 °C, intermediate and low intensity peaks related to the formation of the monoclinic phase of niobium pentoxide were observed. The change in calcination time from 5h to 10h at this temperature caused a reduction in niobium pentoxide to NbO2 and a decrease in crystal size with consequent elevation of the surface area. Oxides calcined at 900 °C presented the largest surface area among those studied in this work.

Acknowledgements:
To UFRN, PPGEQ, CNPq.

References:

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