Supporting Information

A Road to Environmentally Friendly Materials Chemistry: Low-
Temperature Synthesis of Nanosized $\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3$ Powders
Through Peroxide Intermediates in Water

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Contents:

1.) Synthetic charts of KNN nanopowders prepared from pure aqueous solutions…2

2.) IR spectra of Nb(V)-peroxo-based precursors……………………………………………4

3.) IR spectrum of annealed final powders…………………………………………………8

4.) UV-Vis spectra of Nb(V)-peroxo-based precursors……………………………………9
1.) Synthetic Charts of KNN nanopowders prepared from pure aqueous solutions

Fig. 1 Synthesis of KNN powder from Nb(V)-peroxo-citrate aqueous precursor solution

- **30% H$_2$O$_2$ (an excess)**
- **C$_6$H$_8$O$_7$ (citric acid)**
  - 2 : 1 = Citric : Nb
- **pH = 0.80**
- **stirring at 60 °C; 2 hours**

- **(NH$_4$)$_3$NbO(C$_2$O$_4$)$_3$ x H$_2$O**
  - (added in small portions)
- **[Nb(O$_2$)$_4$]$^{3-}$ yellow solution**
- **pH = 8.50**
- **25% NH$_3$(aq)**
  - adding dropwise
- **stirring for an hour**

- **Nb(V)-peroxo citrate**
  - viscous yellow solution

- **CH$_3$COONa (aq)**
  - slowly dropping
- **CH$_3$COOK (aq)**
  - slowly dropping
- **stirring at 60 °C; 4 hours**

- **Alkali Nb(V)-peroxo-citrate aqueous precursor solution**
  - Na : K : Nb = 1 : 1 : 2

- **Water evaporation at 60 °C to obtain dry solid precursor**

- **Calcination at 500 °C for 2 hours**
- **K$_{0.5}$Na$_{0.5}$NbO$_3$ nano-powder**
Fig. 2 Synthesis of KNN powder from Nb(V)-peroxo-glycine aqueous precursor solution

1. Add 30% H$_2$O$_2$ (aq) (an excess) to (NH$_4$)$_3$NbO(C$_2$H$_4$)$_3$ x H$_2$O (added in small portions).
2. Stir at 60 °C for 2 hours.
3. Add H$_2$NCH$_2$COOH (glycine) Nb : glycine = 1 : 2, pH = 1.00.
4. Stir at 60 °C for 3 hours.
5. Add 25% NH$_3$(aq) (adding dropwise), pH = 5.50.
6. Stir at 60 °C for 3 hours.
7. Add NaNO$_3$ (aq) (slowly dropping), KNO$_3$ (aq) (slowly dropping), stirring at 60 °C for 4 hours.
8. Stir at 60 °C for 4 hours.
10. Add K$_{0.5}$Na$_{0.5}$NbO$_3$ nano-powder.
11. Water evaporation at 60 °C to obtain dry solid precursor.
12. Calcination at 550 °C for 2 hours.
2.) IR spectra of Nb(V)-peroxo-based precursors

Fig. 3 FT-IR spectrum of Nb(V)-peroxo-citrate precursor prepared from synthetic protocol presented in Fig. 1.
Fig. 4 Comparison of FT-IR spectra of (a) free citric acid (blue profile) with (b) Nb(V)-peroxo-citrate complex (black profile).
Fig. 5 FT-IR spectrum of Nb(V)-peroxo-glycine precursor prepared from synthetic protocol presented in Fig. 2.
Fig. 6. Comparison of FT-IR spectra of (a) free glycine (blue profile) with (b) Nb(V)-peroxo-glycine complex (black profile).
3.) IR spectrum of annealed final powders

Fig. 7. FT-IR spectrum of K$_{0.5}$Na$_{0.5}$NbO$_3$ nanopowders obtained with annealing of Nb(V)-peroxo-citrate or Nb(V)-peroxo-glycine prepared intermediates at T $\geq$ 500 $^\circ$C for 2-4 hours.

Comment:

- FT-IR spectra of annealed powder samples showed no presence of organic residues (residual carbon) or any other impurities which should have absorption in mid-infrared 4000 – 600 cm$^{-1}$ area.
4.) UV-Vis spectra of Nb(V)-peroxo-based precursors

![UV-Vis spectrum of prepared Nb(V)-peroxo-citrato precursor in water](image)

Fig. 8. UV-Vis spectrum of prepared Nb(V)-peroxo-citrato precursor in water\(^\text{[a]}\)

\(^{[a]}\) strong absorption (\(\sigma \rightarrow d\sigma^*\) LMCT) at 270 nm, weak absorption (\(\pi^\ast \rightarrow d\sigma^*\)) at 387 nm, below 500 nm no absorption was detected.

![UV-Vis spectrum of prepared Nb(V)-peroxo-glycine precursor in water](image)

Fig. 9. UV-Vis spectrum of prepared Nb(V)-peroxo-glycine precursor in water\(^{[a]}\)

\(^{[a]}\) strong absorption (\(\sigma \rightarrow d\sigma^*\) LMCT) at 275 nm, weak absorption (\(\pi^\ast \rightarrow d\sigma^*\)) at 360 nm, below 500 nm no absorption was detected.