

Abstract

The thesis attempts to establish a versatile and technically simple aqueous-based chemical synthesis methodology for the preparation of nanocrystalline powders of CuNb_2O_6 , CuGa_2O_4 , FeNbO_4 , and Pt impregnated FeNbO_4 for gas sensing application and nanocrystalline powders of $\text{Ni}_{0.1}\text{W}_{0.1}\text{Ti}_{0.8}\text{O}_2$, $\text{NiBaTi}_7\text{O}_{16}$, and $\text{Cr}_{2x}\text{W}_x\text{Ti}_{1-3x}\text{O}_2$ ($x = 0.05, 0.1, 0.15$) for pigment application. Nanocrystalline powders of the oxides are produced on calcination of carbonaceous precursors, generated through complete evaporation of the precursor solutions of triethanolamine (TEA) and water-soluble chelated complexes of the desired metal ions.

Gas sensing studies of the compressed pellets of the nanocrystalline powders of CuNb_2O_6 , CuGa_2O_4 , FeNbO_4 , and Pt impregnated FeNbO_4 reveal their sensitivity towards 500 ppm of reducing gases such as, H_2 , liquefied petroleum gas (LPG) and NH_3 . CuNb_2O_6 system shows the maximum response of 84% towards H_2 at 300 °C; around 80% towards LPG at 300 °C; and 56% towards NH_3 at 400 °C, while the CuGa_2O_4 system reveals the maximum response of 82%, 75% and 37% towards H_2 , LPG, and NH_3 respectively at the operating temperature of 350 °C. Similar studies of the FeNbO_4 system exhibit the maximum response of 93% towards H_2 at 250 °C, and 92% and 52% towards LPG & NH_3 gases respectively at 300 °C. Incorporation of platinum into FeNbO_4 (i.e., Pt- FeNbO_4 system) is found to improve the system's gas sensing properties through reduction in the sensor operating temperature, sensor response time and recovery time with maximum sensitivity of 94% towards H_2 at 175 °C, 95% towards LPG at 200 °C, and 61% towards NH_3 at 250 °C.

Ni^{2+} or Cr^{3+} doped nanocrystalline titanate powders [such as: $\text{Ni}_{0.1}\text{W}_{0.1}\text{Ti}_{0.8}\text{O}_2$, $\text{NiBaTi}_7\text{O}_{16}$, and $\text{Cr}_{2x}\text{W}_x\text{Ti}_{1-3x}\text{O}_2$ ($x = 0.05, 0.1, 0.15$)] give rise to interesting color shades in different color range due to $d-d$ transition of the d -electrons of Ni^{2+} or Cr^{3+} ions, and show promising properties for pigments application. The measurement of the color parameters ($CIE L^* a^* b^*$) reflect an intense yellow color with greenish shades for the $\text{BaNiTi}_7\text{O}_{16}$ composition, while the $\text{Ni}_{0.1}\text{W}_{0.1}\text{Ti}_{0.8}\text{O}_2$ solid solution exhibit yellow coloration with reddish shades. Doping Cr^{3+} ion in the rutile lattice on

the other hand, impart buff colouration to the $\text{Cr}_{2x}\text{W}_x\text{Ti}_{1-3x}\text{O}_2$ ($x = 0.05, 0.1, 0.15$) solid solution.

The developed chemical method uses simple metallo-organic based complex chemistry to prepare the aqueous-based precursor solution that contained appropriate amounts of TEA and the desired metal ions in the suitably chelated form. TEA used in the process, not only helps in homogeneous dispersion of the desired metal ions in the precursor solution through formation of stable, water-soluble coordinated complexes with the metal ions but also circumvents sintering of the final powders by generating a porous carbonaceous matrix for the metal ions on dehydration of the precursor solution. Moreover, the carbonaceous precursors (generated from TEA) when calcined generate heat through combustion thereby, facilitates realization of the desired oxide phase at external temperatures much lower than those reported through other routes so far. Low processing temperatures results in nanocrystalline powders of desired oxides with average grain sizes below 60 nm and high specific surface areas up to 110-120 m^2g^{-1} in few cases.