

## Morphology and Properties of Mo Doped Monodispersed Titania Particles

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Titanium dioxide is one of the most efficient photocatalysts for the detoxification of organically charged waste water. However, this material suffers from the drawback of poor absorption properties because of a band gap of 3.2 eV. Thus, wavelengths shorter than 400 nm are needed for light induced generation of  $e^-/h^+$  pairs. Therefore, doping with transition metal ions is interesting for inducing a red-shift of the band gap. However, this doping changes other physical properties such as lifetime of  $e^-/h^+$  pairs and adsorption characteristics. This paper deals with doping titania by  $Mo^{6+}$  ions. Photocatalysis of trichloroethylene with  $TiO_2$  and molybdenum-doped  $TiO_2$  mixed oxide was studied in this work [1] using a tubular quartz reactor packed with photocatalyst-coated glass beads. Nano-sized molybdenum-doped  $TiO_2$  mixed oxide photocatalysts were prepared with the  $Mo^{5+}$  content varying from 0 up to 2.5 mol%, to shift the absorption onset into the visible region and to enhance the efficiency of photocatalytic activity by retarding the  $e^-/h^+$  recombination [2]. In a typical experiment, titanil oxo-sulphate was dissolved in 100 ml of distilled water (1.6 M) and hydrolyzed by slow addition of ammonium hydroxide solution (10%) under constant stirring at temperature of 0 °C in ice bath, until the reaction mixture reaches pH 8.0. The obtained white precipitate was separated by filtration and was washed free of sulphate ions (confirmed by the  $BaCl_2$  test) with distilled water. Obtained wet precipitate was mixed with 100 ml of 30%  $H_2O_2$  solution and yellow gelatinous mass was obtained. The yellow see-through gelatin product obtained by foregoing reaction was mixed with defined amount of ammonium heptamolybdate tetrahydrate  $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$  diluted with 10 ml  $H_2O_2$  (Tab. 1.) and subsequently heated in heating mantle in round-bottom flask with a reflux cooler. During the heating process white precipitate originated. Annealing had been continued till the precipitate changed its color to white or slightly blue (~ 36 hours). The obtained precipitate was dried in the oven at 105°C. HRTEM images of  $Mo^{6+}$  doped anatase particles are shown in Fig 1. The aspect ratio of anatase particle decreased gradually with the addition of molybdenum, and changes the morphology of particles from spindle-like shapes to the shape of rectangular or square cross-section. The HRTEM image confirms that the particles are of anatase structure with the c-axis of the tetragonal crystal collinear with the long axis of the spindle, as indicated by the crystal planes. The fine fringe spacing  $d$  along the [101] direction of the anatase crystal linearly increased with the amount of doping agent. These images clearly show that this is a very good crystalline material, which contains no amorphous domains. The absence of these amorphous domains is an essential prerequisite for good photocatalytic properties.

### References:

- [1] J. K. Yoon, W. S. Yoon et al., J. of the Korean Inst. of Chem. Engineer, 38 (2000) 288-295
- [2] M. S. Jeon, W. S. Yoon, H. Joo, T. K. Lee, H. Lee, Applied Surface Science 165 (2000) 209–216
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Tab. 1. Characteristics of prepared titania samples doped by variable amounts of Mo

Sample	$(\text{NH}_4)_6\text{Mo}_7\text{N}_{24} \cdot 4\text{H}_2\text{O}$ [g]	Crystallite size [nm]	Aspect ratio l/d	BET [ $\text{m}^2\text{g}^{-1}$ ]	Total pore volume [ $\text{cm}^3\text{g}^{-1}$ ]	Cell param. a [Å]	Cell param. c [Å]
TiMo_1	0.05	26.2	3.1	64.4	0.3645	3.79367	9.50879
TiMo_2	0.10	26.2	4.6	100.8	0.2890	3.79467	9.51107
TiMo_3	0.15	45.7	3.6	56.4	0.2925	3.79616	9.51172
TiMo_4	0.25	25.6	2.5	109.4	0.2861	3.79717	9.51300
TiMo_5	0.50	24.2	1.2	107.7	0.3024	3.79870	9.50139
TiMo_6	1.00	15.6	1.0	153.2	0.3007	3.79936	9.50775

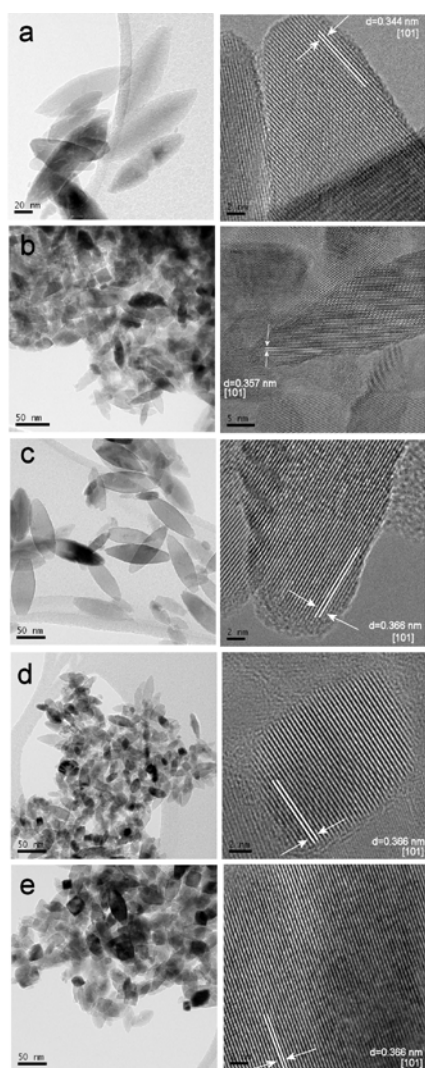


Fig. 1. Molybdenum doped monodispersed titania samples a) TiMo\_1, b) TiMo\_2, c) TiMo\_3 d) TiMo\_4 and e) TiMo\_5