# PRODUCTION OF $SnO_2$ NANO-PARTICLES BY HYDROGEL THERMAL DECOMPOSITION METHOD

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## ABSTRACT

$SnO_2$ is an important functional material having a wide range of applications in gas sensors and optoelectronic devices. There is a great interest for finding new cost-effective and straight-forward methods for production of these particles. In this research, hydrogel thermal decomposition method (HTDM) is used for production of high purity $SnO_2$nano-particles. Cost effective reactants and green routs of production are the advantages of polysaccharide based hydrogel as starting material for this method. Visual observations indicated that there is very little tendency for agglomeration in the $SnO_2$nano-particles produced by this method which can be considered as an advantage for this method over other methods for production of $SnO_2$nano-particles. $SnO_2$nano-particles are also characterized by X-ray diffraction (XRD) in terms of purity and the sizes. It is found that high purity $SnO_2$nano-particles in the size range of 25 – 36 nm can be produced by HTDM.

Key words: nanomaterials, production, $SnO_2$, X-ray techniques, hydrogel thermal decomposition method

## INTRODUCTION

$SnO_2$ is widely used in gas sensors and optoelectronic devices as a functional material [1, 2]. Depending on the production method, $SnO_2$ can be produced with various morphologies [3–8]. Recently, $SnO_2$ with two-dimensional structure has attracted more attention due to its potential applications in gas sensors [9] and photocatalysts [10]. Dai and Pan reported synthesizing $SnO_2$ diskettes by evaporating $SnO_2$ powders at elevated temperatures [11]. Two-dimensional (2D) hierarchical $SnO_2$ were synthesized by Xie et al. [12] using a hydrothermal method based on the reaction between tin foil, NaOH and $KBrO_3$. Hexagonal $SnO_2$nanosheets were synthesized by hydrothermal process using ethanol/ water solution [13]. Flower-like zinc-doped $SnO_2$ nanocrystals have been prepared by a simple hydrothermal process [14]. In addition, $SnO_2$ has been synthesized by template-freemethods [15]. An in-depth study on the productionmethods and the morphological characteristics of the produced $SnO_2$

[2–15] indicates that there is a huge tendency toward finding newcost-effective and straight-forwardmethods for production of these particles.

In the research presented in this article, hydrogel thermal decomposition method (HTDM) is used for production of $SnO_2$ nano-particles. The strategy is the application of starch-graft-poly (acrylic acid) acting as an economical and available effective capping agent. Starch-graft-poly (acrylic acid) with hydroxyl and carboxyl functional groups has a certain binding affinity to metal ions, which may control the size and morphology of $SnO_2$nano-particles without any agglomeration. The feasibility of synthesizing these nanoparticles by HTDM has been verified using XRD.

### *METHODS OF SAMPLE MANUFACTURING AND ANALYSIS*

0.50 g starch, purchased from Merck Chemical Co., was dissolved in 35 ml distillated degassed water. The three-neck reactor was placed in a water bath at 70 C. 0.05 g of potassiumpersulfate (KPS, Merck) as an initiator was added to starch solution and was allowed to stir for 10 min at 70 C. 0.3 g acrylic acid (AA, Merck) and methylenebisacrylamide (MBA, Fluka) solution (0.050 g in 5 ml $H_2O$) were added simultaneously to the starch solution. After addition of monomers, the mixture was continuously stirred (at 200 rpm) for 1 h under argon atmosphere. After 60 min, the reaction product was cooled down to room temperature and neutralized to pH = 8 by addition of 1 N NaOH solution. 500 ml ethanol was added to the gelled product while stirring. After complete dewatering for 24 h, the hardened gel particles were filtered, washed with fresh ethanol and dried at 50°C. A tea bag, i.e., a 100 mesh nylon screen containing 0.1 ± 0.0001 g hydrogel powder was immersed entirely in tin chloride solutions (2000 ppm) and allowed to soak for 48 h at room temperature.

The tea bag was hung up for 15 min in order to remove the excess fluid. A 2000 ppm tin chloride solution was prepared in Erlenmeyer flasks. 0.1 ± 0.0001 g of chelating hydrogel was added to flask. Themixture was shaken for 48 h by a rotary shaker to complete the equilibrium state of reaction. Production line of $SnO_2$nano-particles has been schematically shown in *Figure 1*. $SnO_2$nano-particles were produced by the following typical synthetic procedure. 0.2 g $SnCl_2$ and 0.5 g starch-graft-poly(acrylic acid) hydrogel were suspended in 100 ml water.

The suspension was stirred at room temperature for 48 h. The decantation and freeze drying of the white suspension yielded white powder which was heated at 400 C for 8 h. The final product was washed with water and hexane. X-ray diffraction (XRD) was preformed with a Siemens D5000 X-ray diffractometer using graphite-mono-chromatized high-intensity Cu-Kαradiation ($\lambda$ = 0.15406 nm).

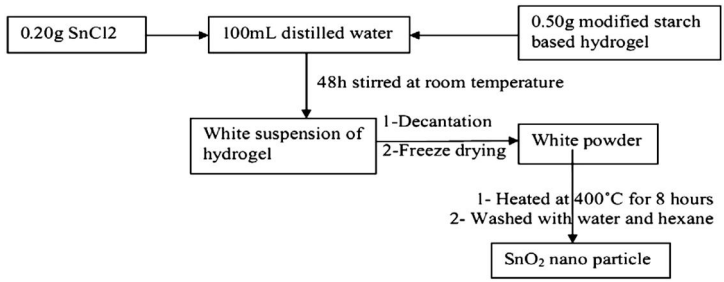


**Fig. 1**– Schematic presentation of the production procedure of $SnO_2$nano-particles.

## *RESULTS AND DISCUSSION*

The produced $SnO_2$nano-particles were investigated by visualobservations for the possibility of agglomeration and by X-raydiffraction method for checking purity and the size range of thenano-particles. Visual observations of the produced $SnO_2$ nanoparticles demonstrated that by HTDM there is very little tendency for agglomeration. This can be counted as an advantage of the HTDMmethod for production of $SnO_2$nano-particles.

XRD patterns of the produced $SnO_2$nano-particles are shown in *Figure. 2*. It is clear that no obvious reflection peaks from the impurities, i.e., other tin oxides, are detected. This indicates that the HTDM results in a product with high purity which yields an effective procedure for synthesis of pure $SnO_2$nano-particles. The Miller indices are indicated on each diffraction peak. The diffraction peaks of the (110), (101), (200), (211), (220), (002), (310), (112), and (301) planes can be readily indexed to the tetragonal structure of SnO2 with lattice constants of a = 4.738 Å and c = 3.187 Å (JCPDS File No. 41–1445).

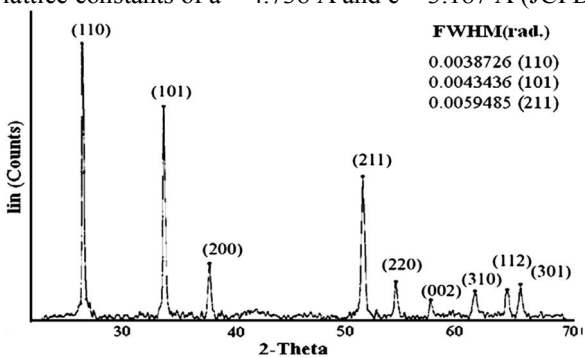


**Fig. 2**– XRD patterns of the $SnO_2$nano-particles

The mean particle size was calculated for the $SnO_2$nano-particles using the Scherer's equation, Eq. (1), from the reflections with (110), (101) and (211) indices

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

Where D is the mean size of particle (nm), k, a constant (0.89), λ, the X-ray wavelength (0.15405 nm), β, the full-width at half-maximum (FWHM radian) of XRD peaks and θ, the Bragg's angle (deg).

The mean size of nano-particles was calculated in the range of 25 to 36 nm. This value was in good agreement with TEM observation of the $SnO_2$nano-particles. This indicates that production of finer $SnO_2$nano-particles with high purity is the significant advantage of synthesizing by HTDM. The results demonstrated that the presented hydrogel in this study may serve as a platform for production of tin oxide nano-particles. Low-priced reactants and green routs for synthesis are the advantages of polysaccharide based hydrogel as starting material for this method.

### *CONCLUSIONS*

In this research, the feasibility of synthesizing $SnO_2$ nano-particlesby HTDM was investigated. The sizes and purity of the nano-particleswere investigated by X-ray diffraction method. It was found that thepresented hydrogel in this study may serve as a platform for production of tin oxide nano-particles. According to the results ofthe current investigation the following conclusions were made:

1. – Production of high purity $SnO_2$nano-particles with a size range of 25–36 nm is possible by application of the HTDM method. Lowpricedreactants and green routs for synthesis are the advantages of polysaccharide based hydrogel as starting material for this method.

2. – There is very little tendency for agglomeration in the $SnO_2$ nanoparticles produced by HTDM. This can be considered as an advantage for this method over many other methods for production of these particles.

### *REFERENCES*

[1] T. Sahm,W. Rong, N. Barsan, et al. Sens. and Actuat. B,2007,127:63–8.
[2] N.N. Samotaev, A.A. Vasiliev, B.I. Podlepetsky, A.V. Sokolov, A.V. Pisliakov. Sens. andActuat., B, 2007,127:242–7.
[3] H. Wang, J. Liang, H. Fan, et al. J. Solid State Chem., 2008, 181:122–9.
[4] Z.R. Dai, Z.W. Pan, Z.L. Wang. Solid State Commun., 2001, 118:351–4.
[5] W.Z. Wang, J.Z. Niu, L. Ao. J Cryst Growth, 2008, 310:351–5.
[6] T. Krishnakumar, N.K. Pinna, P. Kumari et al.. Mater Lett., 2008, 62:3437–40.
[7] E.R. Leite, I.T. Weber, E. Longo, J.A. Varela. Adv Mater., 2000,12:965–8.
[8] C. Nayral, T. Ould-Ely, A. Maisonnat et al. Adv. Mater.,1999, 11:61–3.
[9] J.P. Ge, J. Wang, H.X. Zhang, X. Wang, Q. Peng, Y.D. Li. Sens.andActuat. B, 2006,113:937–43.
[10] W.W. Wang, Y.J. Zhu, L.X. Yang. Adv.Funct.Mater., 2007,17:59–64.
[11]Z.R. Dai, Z.W. Pan, Z.L. Wang. J. Am. Chem. Soc., 2002,124:8673–80.
[12]Q.R. Zhao, Z.Q. Li, C.Z. Wu, B. Xue, Y.J. Xie. Nanopart. Res., 2006,8:1065–9.
[13]X.H. Yang, L.L. Wang. Mater.Lett., 2007;61:3705–7.
[14]Z.R. Li, X.L. Li, X.X. Zhang, Y.T. Qian. J.Cryst.Growth., 2006, 291:258–61.
[15]Y. Li, Y. Guo, R. Tan, P. Cui, Y. Li, W. Song. Mater.Lett., 2009, 63:2085–8.