A NOVEL METHOD OF PREPARING AND FIXING TiO_2 NANOMETER PARTICLES

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It is well known that during the past two decades, TiO_2 has become an important subject.[1] But its separation and recollection have limited its further commercial applications, especially in photoelectro-chemistry field[2]. Thus, its preparation has become a key problem in its recent study. Tetrabutyl titanate has been used as materials in many TiO_2 synthesis methods,[3] but obviously, it is too expensive comparing with titanium tetrachloride we introduced and employed in this paper. To the best of our knowledge, for the preparation of TiO_2, there is no paper reporting the method carried out in liquid phse and ambient pressure, where titanium tetrachloride was utilized to low down the cost.

The aim of this paper is to show a low cost method of preparing different structures TiO_2 nanometer particles, which was performed in liquid and ambient pressure. Encouragingly, the novel TiO_2 nanoporous film electrode produced from these TiO_2 nanometer particle exhibited satisfactory results in the photo-electro oxidation of methanol, which is meaningful to widen TiO_2 application.

Experimental

1. The first step is to prepare $TiOCl_2$ solution using $TiCl_4$. Transferring TiCl4 in the icy distilled water firstly, after stirring for several hours completed this step, $TiOCl_2$ solution was obtained. And then transferring a part of this solution into 150ml flask, simultaneously, some additives containing SO_4^{2-} were introduced into the flask, where the boiling refluxing process at least proceeded for 5 h. After extracting, washing and desiccating, the anatase TiO_2 nanometer particles ranging from 10 to 50 nm of particle diameter were obtained as shown in figure1. Interestingly, when no additives were introduced, mixed structure TiO_2 nanometer particles with 50nm could be produced as shown in figure 1f, hinting that the types of additive could determine the particle diameter of TiO_2 partly. What's more, when the ageing time is 48 h, rutile configuration TiO_2 nanometer particles were obtained, as shown in figure 1a, suggesting that different ageing time could lead to TiO_2 nanometer particles with different configuration.

2. The fixation of TiO_2 nanometer particles. After transferring above 6 kinds TiO_2 nanometer particles into 50 ml flask, respectively, some distilled water was introduced into the flask, which was followed by 1h's supersonic dispersing. And then some kinds of polyethylene glycol were added, accompanying by stirring for 30 min. The last step is to transfer the white sol onto the conducting glass by dipping the conducting glass, after sintering at 400 °C for 30 min, the TiO_2 nanoporous film electrode was obtained.

3. The photoelectro-chemical property of TiO_2 nanoporous film electrode. The latest generated TiO_2 nanoporous film electrode was used in the photoelectrochemical oxidation of methanol, which was also a focus in solar cell investigation. In contrast, the photoelectrochemical oxidation of methanol could be regarded as a probing pin to reflect the characteristic of TiO_2 nanometer particle, as was illustrated in table 1. The anatase TiO_2 nanoporous film electrode exhibits the maximum photocurrent under the same particle diameter. Meanwhile, for anatase TiO_2 nanoporous film electrode, the photocurrent was increased by several times when the diameter was decreased., which probably was caused by the augment of surface area.

Conclusions

The main contribution of this paper is have developed a novel method to prepare the different structure TiO_2 nanopmeter particles, which was performed in liquid phase and ambient pressure. What's more, the new generated TiO_2 nanoporous film electrode was also satisfactory, which is helpful to explore the new application of TiO_2 nanometer particles.

References

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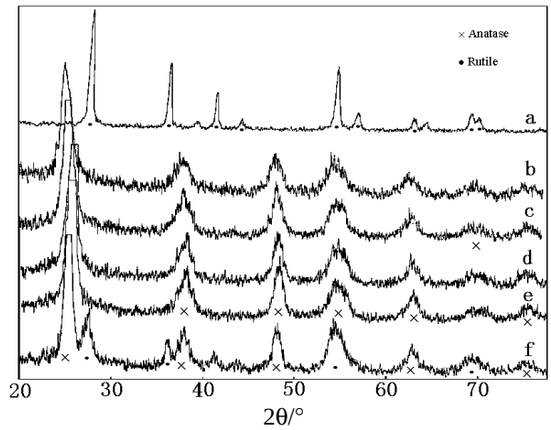

Fig.1. XRD patterns of different TiO_2 nanometer particles with different crystal structures, shapes, particle diameters

Table 1. Photocurrents of different TiO_2 nanoporous film electrode

No.	crystal structure	shape	diameter (nm)		I_{ph} mA cm^{-2}
			SEM	XRD	
a	Rutile	Rhomb	~50		0.13
b	Anatase	Sphericity	~50	44	0.15
c	Anatase	Sphericity	~25	30	0.4
d	Anatase	Sphericity	~20	21	0.5
e	Anatase	Sphericity	~15	16	0.54
f	Mixed	Sphericity	~55	52	0.05