Aqueous and non-aqueous sol-gel preparation of TiO₂ films for the photocatalytic oxidation of ethanol in air

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Introduction

Photocatalysis is successfully applied to environmental remediation. The photocatalytic activity of TiO₂, as well as its physico-chemical stability, self-cleaning property and high selectivity make it a suitable candidate for the removal of hazardous pollutants such as VOCs from contaminated indoor atmosphere.

The sol-gel process is widely used to obtain porous and homogeneous TiO₂ and it allows to control the stoichiometry and work in mild and ambient atmospheric conditions.

The aim of this study was to synthesize TiO₂ sols by two different sol-gel methods (aqueous and non-aqueous) and to evaluate the influence of different solvents and different concentrations of hydrolyzing agent on the photocatalytic activity of the films.

Aqueous sol-gel

TiO₂ sols were synthesized using titanium tetraisopropoxide (TTIP) as precursor and nitric acid in water as hydrolyzing agent.

Three different solvents (ethanol, isopropanol and butanol) and two different molar ratios HNO₃/Ti (0.25 and 0.5 respectively) were used in order to evaluate the best film preparation conditions (films A-F).

Films produced by consecutive spin coating of 4 layers were heat treated at 450°C for 2 hours and tested in the breakdown reaction of ethanol as VOC molecule under UV and visible light.

Photocatalytic measurements were carried out in a stainless steel batch reactor, in a controlled Ar/O₂ atmosphere, by means of an atmospheric gas analyser containing a mass spectrometer [1].

Non-aqueous sol-gel

Titanium tetraisopropoxide was used as precursor and the hydrolysis was initialized by esterification reaction between acetic acid and ethanol. Acetylacetone was added as chelating agent (film G).

Results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Solvent</th>
<th>HNO₃/Ti precursor ratio</th>
<th>dXRD (nm)</th>
<th>SAXRD (m²/g)</th>
<th>SA BET (m²/g)</th>
<th>dBET (nm)</th>
<th>Photocatalytic activity (ppm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>EtOH</td>
<td>0.25</td>
<td>63</td>
<td>25</td>
<td>&lt; 5</td>
<td>-</td>
<td>1.84</td>
</tr>
<tr>
<td>B</td>
<td>EtOH</td>
<td>0.5</td>
<td>37</td>
<td>41</td>
<td>&lt; 5</td>
<td>-</td>
<td>2.67</td>
</tr>
<tr>
<td>C</td>
<td>iPpOH</td>
<td>0.25</td>
<td>60</td>
<td>25</td>
<td>&lt; 5</td>
<td>-</td>
<td>0.25</td>
</tr>
<tr>
<td>D</td>
<td>iPpOH</td>
<td>0.5</td>
<td>36</td>
<td>42</td>
<td>19</td>
<td>76</td>
<td>1.32</td>
</tr>
<tr>
<td>E</td>
<td>BuOH</td>
<td>0.25</td>
<td>151</td>
<td>10</td>
<td>50</td>
<td>30</td>
<td>1.55</td>
</tr>
<tr>
<td>F</td>
<td>BuOH</td>
<td>0.5</td>
<td>62</td>
<td>25</td>
<td>23</td>
<td>67</td>
<td>1.78</td>
</tr>
<tr>
<td>G</td>
<td>EtOH</td>
<td>—</td>
<td>90</td>
<td>18</td>
<td>35</td>
<td>44</td>
<td>1.06</td>
</tr>
<tr>
<td>P25</td>
<td>—</td>
<td>—</td>
<td>25</td>
<td>61</td>
<td>56</td>
<td>27</td>
<td>3.44</td>
</tr>
</tbody>
</table>

Conclusions

- A higher concentration of HNO₃ led to smaller crystallite size and hence to a higher photocatalytic activity.
- The aqueous sol-gel films, except for sample C, showed a higher photocatalytic activity than the non-aqueous sol-gel film.
- The aqueous sol-gel process in which ethanol has been used as solvent (samples A-B), led to highly active films if compared with TiO₂ films prepared from Degussa P25.

References


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