Cost-Effective Teacher

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An Inexpensive Furnace for Calcination: Simple TiO_2 Synthesis

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Calcination is a thermal process applied to solid materials to effect a thermal decomposition, a phase transition, or removal of a volatile component. The process often requires an expensive furnace or reactor that can operate at temperatures below the melting point of the material. Many materials, such as activated carbon, metal oxide, hydroxyapatite, and so forth, are calcined at temperature below 500 °C *(1–3).* An alcohol burner and a Bunsen burner, normally found in any laboratory, have a working temperature of 350–500 °C, which can be suitable for calcination.

An increasing number of researchers are studying titanium dioxide (TiO_2) as verified by the doubling of Chemical Abstract citations involving TiO_2 between 2000 and 2006. This *Journal* has also published experiments that use TiO_2 as a material to help students understand the concepts of photocatalysis and dye-sensitized solar cells *(4–11).* For these reasons, TiO_2 nanoparticle synthesis has been selected to demonstrate the calcination process using an alcohol burner. In this article, we describe a way to synthesize TiO_2 nanoparticles with a high degree of crystallinity using a simple and inexpensive ethanol burner.

Equipment and Method

Ethanol Burner

A simple ethanol burner setup for the calcination process is shown in Figure 1. The setup is composed of an alcohol burner, clamp, stand, porcelain crucible, and a plate shielding. A crucible is clamped to the stand. The distance between the bottom of crucible to the top of a lamp wick is ~2 cm. The length of the lamp wick is ~1 cm. The temperature inside the crucible measured with a Center 350 infrared thermometer fluctuates from 450 to 480 °C.

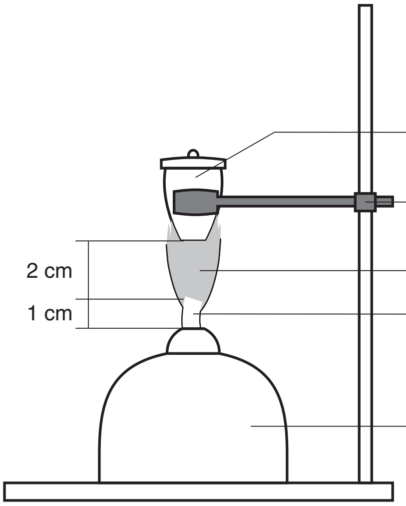

Figure 1. (left) A simple ethanol burner for the calcination process and (right) sketch of the simple ethanol burner setup.

Synthesis of TiO_2 Nanoparticles

Titanium(IV) isopropoxide (TTIP; Fluka) was used without further purification as the starting material for the synthesis of TiO_2 nanoparticles. TTIP, 20 mL, was added dropwise to 61.2 mL of distilled water (molar ratio of TTIP:H_2O is 1:50) with vigorous stirring at 1000 rpm for 20 minutes. The precipitate was then dried at 120 °C for 10 hours. The powder obtained was manually ground for 10 minutes and subsequently placed in the porcelain crucible and calcined using an ethanol burner for an hour *(12–15)*. The synthesis process is summarized as a flowchart in Figure 2. Other fuels can be substituted for ethanol: methanol, propanol, or commercial camping fuel (propane:butane, ~40:60 v/v).

Characterization

The TiO_2 powders were characterized by an X-ray powder diffractometer (XRD, Bruker D8 Advance 40 kV, 40 mA) using Cu Kα radiation as the source. Diffractograms, 2θ, were collected from 20 to 60° with a step size of 0.0157311° and a step time of 0.6 s/step.

The morphology and grain size of the TiO_2 powders were investigated by transmission electron microscopy (TEM) on a Tecnai G2 Sphera operating at 80 keV. TEM samples were prepared by dipping a carbon-coated Cu grid in a 100 mg L^{-1} TiO_2 suspension several times and dried at ambient temperature. Over 20 configurations of each sample were investigated.

The Brunauer–Emmett–Teller (BET) surface areas of the powders were determined by nitrogen adsorption at −196 °C using a Quantachrome Autosorb 1 sorption analyzer. The samples were degassed at 300 °C for 3 hours prior to nitrogen adsorption measurements.

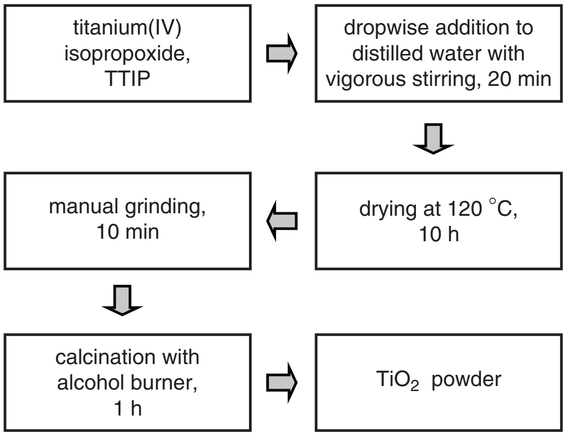

Figure 2. Flowchart of the synthesis procedure.

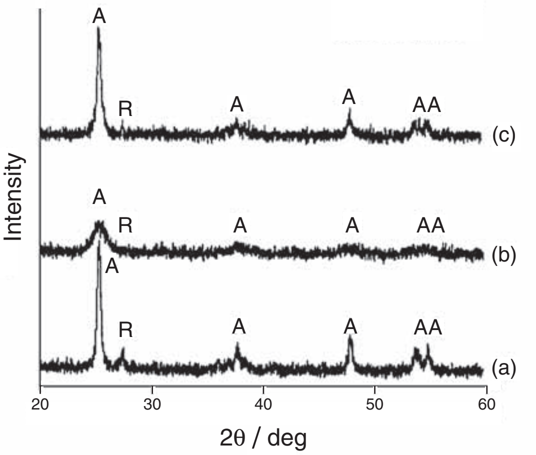

Figure 3. X-ray diffractograms of the TiO_2 powders: (a) commercial Degussa, P-25, (b) synthetic product before calcination, and (c) synthetic product after 1 hour of calcination. The anatase and rutile phases are labeled with an A and R, respectively.

Results and Discussion

The X-ray diffractograms of TiO_2 powders are shown in Figure 3. The spectrum of the commercial TiO_2 nanoparticles, Degussa, P-25 (Figure 3a), is used as the standard. Before calcination (Figure 3b), the prepared TiO_2 peaks were broad and of low intensity indicating little degree of crystallinity with an anatase phase (2θ = 25.25°). After calcination for an hour with the ethanol burner (Figure 3c), the particle size and crystallinity of the anatase phase increased, as shown by the narrower peaks and higher peak intensity. Crystallinity and particle size are known to increase with increasing calcination time until all phase transformation is completed *(15)*. These diffractograms demonstrate that calcination with the ethanol burner can yield crystalline TiO_2 of same quality as the commercial TiO_2 nanoparticles.

The TEM micrographs of commercial TiO_2 nanoparticles and the calcined TiO_2 nanoparticles are shown in Figure 4. The average particle size of the calcined TiO_2 powder is 15 ± 5 nm compared to 25 ± 8 nm for the commercial TiO_2 nanoparticles *(16, 17)*. The BET specific surface areas of the synthetic and the commercial samples are 75 m^2 g^{-1} and 63 m^2 g^{-1}, respectively. The TEM particle size and BET results are in good agreement. The calcined TiO_2 powder has a higher surface area compared to commercial TiO_2 powder, which correspond to the results of the particle size from TEM.

Hazards

Ethanol is highly flammable. Titanium(IV) isopropoxide is irritating to the eyes, skin, and respiratory tract and is flammable. Students should wear resistant gloves and goggles. Samples should be manipulated in a well ventilated fumehood.

Conclusions

A simple alcohol burner can be used to replace an expensive furnace in the calcination process with temperatures below 500 °C. As a case study, X-ray diffractogram, TEM micrographs, and BET surface area analysis of the synthetic TiO_2 show that the TiO_2 powders obtained by this simple inexpensive calcination process can yield TiO_2 powder with comparable degree of crystallinity, smaller particle size, and higher specific surface area compared to commercial TiO_2 nanoparticles.

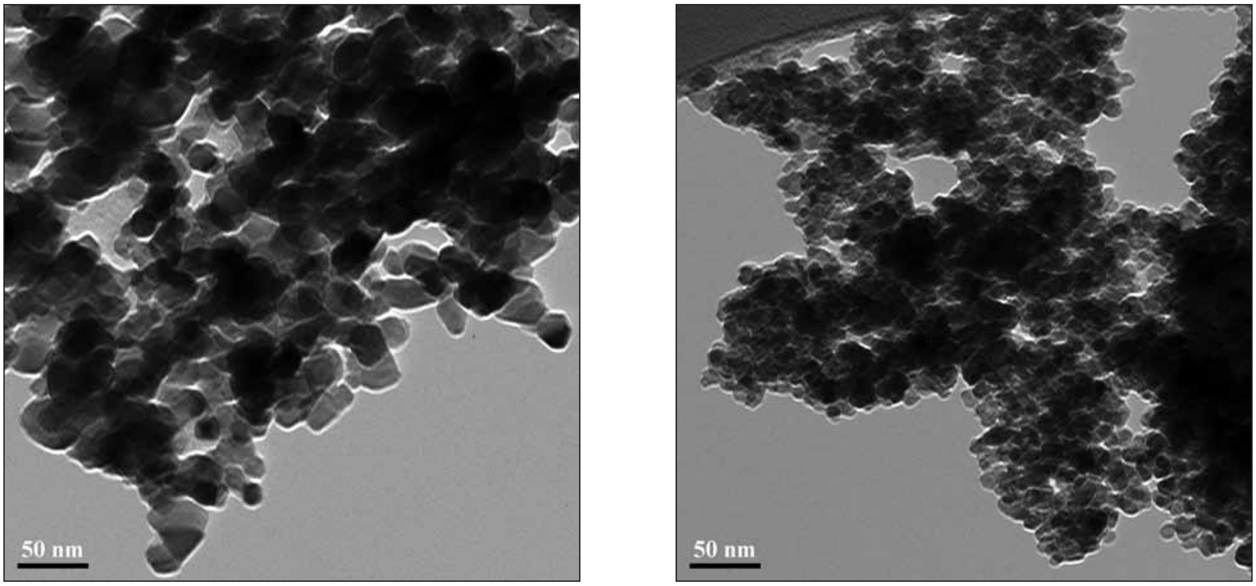

Figure 4. Typical TEM micrographs of the TiO_2 powders: (left) commercial Degussa, P-25 and (right) synthetic product after 1 hour of calcination.

Acknowledgments

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