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TiO2 Coating Method for Nanocellulose Aerogel

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Aerogel Creation

Nanocellulose aerogels were created using a mechanical process which used an ultrasonic probe to break apart the microcrystalline cellulose into smaller particles and nano fibers creating a suspension in water. The suspension was then placed under vacuum and freeze dried to remove the liquid leaving the nanocellulose aerogel. Titanium isopropoxide purchased from Acros Organics was the source material for coating the nanocellulose aerogels in a layer of titanium dioxide. Multiple processes were studied in order to find the best coating method. Figure 1 provides SEM images of coated and uncoated aerogels using method C.

Coating Methods

A) Nanocellulose aerogels were submerged in titanium isopropoxide for 30 seconds and set out to air dry. This method was good in insuring all of the aerogel was coated with titanium isopropoxide, but many of the aerogels compressed and lost their volume when submerged in the liquid. Losing shape creates a material that does not have the aerogel characteristics needed for increased hydrogen production efficiency.

B) Nanocellulose aerogels were placed in vapor given off by the titanium isopropoxide for 12 hours. Figure 2 shows the setup with the beaker on a 150°C hot plate. After, the aerogels were submerged in water for 10 seconds to create the layer of titanium dioxide. The titanium isopropoxide reached the insides of the aerogel creating a layer of titanium dioxide, but submerging the nanocellulose aerogel in water collapsed the aerogel structure needed to be a good catalyst.

C) The Nanocellulose aerogels were placed in the titanium dioxide vapor for 12 hours. The aerogels were then set out to dry in the air. This method proved to be the most consistent in limiting the change in volume and creating even titanium dioxide coatings. The titanium isopropoxide vapor reached the inside of the aerogel creating a layer of titanium dioxide while still keeping the aerogel shape and volume.

In all three methods, water is needed to create the layer of titanium dioxide. In method A and C, the water comes from atmospheric humidity. In method B, the water and titanium isopropoxide reaction occurs when the nanocellulose aerogel is submerged in water for 10 seconds. The reaction of water and titanium isopropoxide is

\[
\text{Ti(OCH(CH_3)O)-fetch + 2H_2O \rightarrow TiO}_2 + 4(\text{CH}_3)_2\text{CHOH} 
\]

with isopropanol being the product along with titanium dioxide. The isopropanol evaporates leaving behind the titanium dioxide coating.

Results

- Method C proved to be the best method in conserving the aerogel shape and producing an even titanium dioxide coating (See Table 1 for densities)
- EDX showed that 12 or more hours was enough to fully coat the inside of the aerogel
- Aerogels with a lower concentration than 5% had too significant of a decrease in volume and were deemed unusable
- The resulting densities are very similar. This suggests that when coated with titanium dioxide, the nanocellulose aerogels all dry out to the same density no matter the initial density

After the titanium dioxide coating, the surface area of the 10% aerogel increased from 8.7 m²/g to 315.8 m²/g. Crystals typically grow in a structure that limits surface area while maximizing volume, so an increase this big was not expected. The density of nanocellulose alone is 1.46 g/mL and the density of titanium dioxide alone is 4.23 g/mL. The 10% coated aerogel's density increased by a factor of 5 indicating there are equal parts nanocellulose and titanium dioxide. The 5% aerogel density increased by a factor of 10 indicating there is a larger volume ratio of titanium dioxide to nanocellulose after coating.

Further Analysis

The next step is to see how well this new material aids in the decomposition of pollutants or the splitting of water. Also, rutile titanium dioxide was used by Fujishima and Honda in 1972 when they first discovered the ability for titanium dioxide to split water. We believe that we are using primarily anatase titanium dioxide. X-ray diffraction spectroscopy will be needed in order to determine the correct crystalline structure we have. Once determined, we will test the effectiveness as a catalyst.

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