

## TEM–XRD analysis of PdO particles on TiO<sub>2</sub> support for chemochromic detection of hydrogen

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### ABSTRACT

Safety is always a concern in all applications that utilize hydrogen (H<sub>2</sub>) in one form or another. Hydrogen leaks are invisible and odorless. In addition, blending odorants or additives into hydrogen in a manner similar to natural gas is generally undesirable for certain applications, including proton exchange membrane fuel cells. To facilitate detection of the location of hydrogen leaks, a special nonreversible chemochromic H<sub>2</sub> sensing material that employs titania (TiO<sub>2</sub>) supported palladium oxide (PdO) pigments encapsulated within a special silicone matrix has been developed at the Florida Solar Energy Center (FSEC) and field tested at National Aeronautics and Space Administration, Kennedy Space Center. Several batches of PdO hydrogen gas sensing pigments were synthesized using various TiO<sub>2</sub> supports, and their hydrogen detection activity was determined. TEM and particle size distribution analysis showed that smaller particles with a hemispherical, crystalline structure produced faster coloration kinetics when exposed to H<sub>2</sub> gas. However, agglomerated PdO particles on the TiO<sub>2</sub> surface displayed greater color contrast. XRD analysis indicated that the crystalline phase of TiO<sub>2</sub> had no effect on the chemochromic performance of the pigments in a laboratory environment.

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### 1. Introduction

Hydrogen (H<sub>2</sub>) is an important chemical commodity produced and used in many industries such as the reduction of metal oxides (e.g., iron ore), ammonia synthesis, and production of hydrochloric acid, methanol and higher alcohols, aldehydes, hydrogenation of various petroleum, coal, oil shale and edible oils, among others. Furthermore, hydrogen is a prospective energy carrier poised to replace fossil-based transportation fuels and currently is the primary fuel of today's space vehicles (e.g., rocket motors). It is also used in fuel cells that generate electrical power. One key issue, in all hydrogen-related applications, is operational safety. Hydrogen is a colorless, odorless gas with a lower explosive limit of about 4% in air. Therefore, reliable and rugged sensors are required to detect hydrogen leaks wherever it is produced, stored, or utilized. Current hydrogen sensors typically need a dedicated operator to monitor the instrument and are also susceptible to environmental interferences in open areas.

In this paper, we describe the development of a simple and robust nonreversible chemochromic hydrogen sensing material that can be readily applied to the surface of the hydrogen transport pipes, flanges, joints, and all hard to reach places that may encounter the possibility of the hydrogen gas leaks. The technique provides a visual method for detecting and locating H<sub>2</sub> leaks especially during hazardous handling and operations when personnel cannot be present, as it does not require any power or operator presence. This is especially important during H<sub>2</sub> transportation, loading, and storage, due to the low flammability limit of hydrogen.

TiO<sub>2</sub>-supported PdO hydrogen sensing pigments described in this work, when exposed to H<sub>2</sub> gas, undergo a color change transformation from beige to dark gray or black color depending on PdO concentration. This transformation is mainly due to PdO reduction by hydrogen to metallic Pd (Eq. (1)) [1]. This paper presents the experimental results for the effect of PdO particle size and distributions over various TiO<sub>2</sub> on the overall chemochromic behavior of the hydrogen sensing pigments.



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## 2. Materials and methods

### 2.1. Pigments preparation

Using a modified procedure reported by Sakamoto et al. [2], several chemochromic pigments were synthesized by using four different TiO<sub>2</sub> support materials: Aldrich titania (mainly, rutile crystalline form) with an average particle size of 1 μm, Fisher Scientific TiO<sub>2</sub>, DuPont R103 TiO<sub>2</sub>, and Degussa P25 nanosize TiO<sub>2</sub>.

Pigments having 3 wt% Pd were prepared by dissolving 1.0 g of PdCl<sub>2</sub> in 50 mL of 2 M Hydrochloric acid (HCl) solution. Two syringes were filled, one with 30 mL of PdCl<sub>2</sub> solution and the other with 30 mL of 2 M NaOH solution. Both syringes were placed in a programmable syringe pump, and the injection rate was set to 0.5 mL/min. Then, a 25 mL of each solution was added to a TiO<sub>2</sub> slurry (10 g TiO<sub>2</sub> in 100 mL H<sub>2</sub>O) heated to 70 °C at a pH of 10. During this process, solution pH remained within the 10–11 (±0.5%) range. The solution pH was then adjusted to 8 by adding 3 M HCl solution. The solution was stirred for an hour and then filtered, washed, and oven dried at 110 °C.

### 2.2. Tape preparation

The pigments alone, in their powder form, are not useful in harsh conditions. Hence, an encapsulation technique in a silicone matrix has been developed to overcome some of the limitations such as being washed out or interference from other reducing gases. This matrix has a high permeability toward hydrogen, is flexible and hydrophobic, and has low permeability of water vapor and other reducing gases such as carbon monoxide. The chemochromic hydrogen sensing tape, used in this study, was a mixture of 3 wt% of pigment in DOW Corning® RTV 3145 silicone matrix. Typically, the mixture was spread into thin films (approximately 2 mils) using a Gardco adjustable film applicator on wax paper.

### 2.3. Transmission Electron Microscopy (TEM)

An FEI/Philips, Tecnai F30, 300 kV field emission source TEM instrument, equipped with STEM, an HAADF detector, and XEDS was used to perform TEM bright field high resolution imaging analysis. The software used was Tecnai G2 Digital Micrograph (DM) and Tecnai Imaging & Analysis (TIA). Samples were mounted on Quantifoil holey carbon grids by placing a few droplets of a methanolic suspension of the pigment, followed by air drying.

### 2.4. X-ray diffraction (XRD)

The crystalline phase of the TiO<sub>2</sub> particles was analyzed by means of Rigaku XRD using a CuK<sub>α</sub> radiation at 40 kV from 10° to 80° at a rate of 2° min<sup>-1</sup> (2θ).

### 2.5. XPS analysis

XPS analysis of the hydrogen sensing pigment prior and after hydrogen exposure was conducted on a Kratos XSAM 800 spectrometer at a background pressure of 1 × 10<sup>-9</sup> torr, using a Mg K<sub>α</sub> (hν = 1253.6 eV) X-ray source. The X-ray beam used was 150 W, 4–6 mm in diameter. The collected data were referenced to the C 1s peak to 284.6 ± 0.5 eV. Wide survey scans were collected from 0 eV to 1100 eV at a pass energy of 80 eV in 1 eV steps with a 50 ms dwell time to determine overall elemental composition. High resolution scans of the C 1s, O 1s, Pd 3d, and Ti 2p peaks were acquired at a pass energy of 20 eV, in 0.1 eV steps with a 300 ms dwell time to determine the chemical state. The relative atomic concentrations of the detected elements were calculated and normalized to 100%

using sensitivity factors [3] supplied by the instrument manufacturer from known certified standards.

### 2.6. Color change measurements

The chemochromic effects of the membranes were evaluated by placing them in a hydrogen exposure chamber. The volume of the chamber was approximately 20 mL, and the flow through the chamber was approximately 50 sccm. After exposure, a ColorTec-PCM colorimeter was used to measure pigments color change before and after exposure to hydrogen gas. Background interference was minimized by placing the samples on a black surface prior to all measurements.

### 2.7. Environmental exposure test

To test the durability of the chemochromic tapes, they were subjected to tests in the laboratory and outdoors at the Kennedy Space Center (KSC) corrosion beach site. At the beach site, tapes were exposed to atmospheric conditions located approximately 100 feet from the Atlantic Ocean. Individual pieces were attached to a stainless steel panel, and the panels were placed on racks. The racks are 5 feet high and oriented 60° from the plumb line. Laboratory studies included submerging the tape in deionized (DI) water and in a 3.55% sodium chloride (NaCl) solution for 12 days. The tapes were also placed in an oven set at 95 °F and exposed to UV light for 12 h. The UV light was inserted into a box lined with aluminum foil to maximize the intensity of the radiation.

## 3. Results and discussions

### 3.1. Pigments' responses to hydrogen exposure

As previously noted, when TiO<sub>2</sub>-supported PdO pigments are exposed to hydrogen gas, PdO is reduced to elemental Pd resulting in a color change from beige to dark gray. These pigments have to be immobilized on the surface of interest, by first being mixed with a resin and applied either as a paint or tape. In this study, DOW Corning® RTV 3145 silicone was chosen to be mixed with pigment and then cast as tape. Silicones are known to have very high hydrogen solubility [4] and hence are the best candidate for pigment application when any environmental factor such as wind and rain is present.

Pigments' responses to H<sub>2</sub> exposure were measured by measuring the extent of the color change by a colorimeter that incorporates an algorithm capable of quantifying the color change, ΔE, using following parameters: *L*—lightness value, *a*—position on red–green axis, and *b*—position on yellow–blue axis.

$$\Delta E = \{(L - L')^2 + (a - a')^2 + (b - b')^2\}^{1/2} \quad (2)$$

Eq. (2) gives a standard measurement with which to compare the color change associated with different samples. The chemochromic tapes were analyzed before and after exposure to hydrogen gas, allowing quantification of the extent of color change. Other techniques such as UV–vis could also be used to measure this color change, but since this sensor will be mainly monitored visually, a technique more suitable for human eye is more desirable.

Each pigment formulation was exposed to pure hydrogen in an exposure chamber and analyzed at specific time intervals. Samples were analyzed with the colorimeter to obtain the corresponding ΔE value. Fig. 1 depicts the ΔE values vs. hydrogen exposure time for four different titania supports in the TiO<sub>2</sub>/PdO pigments. All pigments started to change color after 1.5 min of exposure time except the Degussa P-25 based pigment, which began changing color almost immediately after exposure to H<sub>2</sub> gas and reached

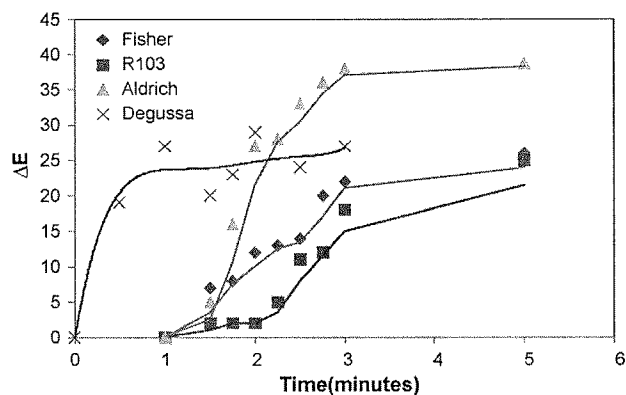


Fig. 1.  $\Delta E$  vs. exposure time to  $H_2$  for four different titania supports in  $TiO_2/PdO$  pigments.

saturation after 1 min. The pigment based on Aldrich  $TiO_2$  demonstrated the highest value of  $\Delta E$  ( $=38$ ) after 3 min of exposure time, while other pigments reached  $\Delta E$  values no more than 26 for the same length of exposure time.

Preliminary tests were conducted to determine response to low levels of hydrogen. With the flammability limit of 4% hydrogen, the four chemochromic tapes were exposed to the 1% hydrogen in argon gas mixture for 2 min and calorimeter readings were taken (Fig. 2). Note that color change kinetics can greatly be affected by the membrane thickness, and hence the kinetic data presented in Figs. 1 and 2 are for 2 mil thick samples and only to compare various pigments. The kinetics of color change for virgin pigment as a powder will be shortly submitted for publication.

### 3.2. XRD

Most  $TiO_2$  photocatalysts contain an anatase crystalline phase [5]. Anatase titania has a band gap of 3.2 eV, while rutile  $TiO_2$  has a band gap of 3.0 eV. Rutile  $TiO_2$  has limited photocatalytic activity. Fig. 3 shows the XRD patterns for  $TiO_2/PdO$  pigments prepared in this study. The sharp peaks at  $25.3^\circ$  and  $47.9^\circ$  were attributed to anatase  $TiO_2$  [6–9] which is the dominant phase for Degussa P-25 and Fisher  $TiO_2$ . Aldrich and DuPont R103  $TiO_2$  are mainly composed of rutile phase. These results indicate that photocatalytic activity of  $TiO_2$  or lack thereof has no effect on the chemochromic activity of  $TiO_2/PdO$  pigments in the laboratory environment. Furthermore, all samples with the exception of pigment based on Fisher  $TiO_2$ , contain a small peak at  $32.6^\circ$  attributed to the reflection of PdO.

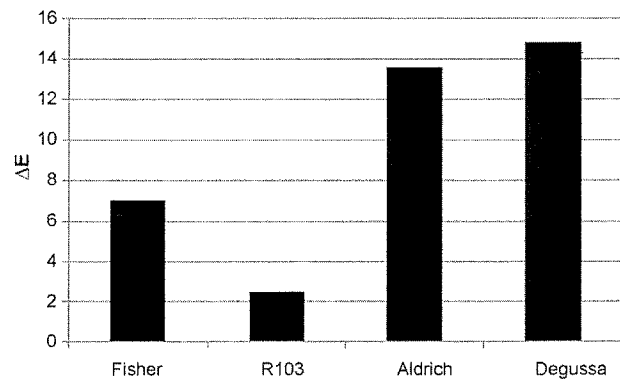


Fig. 2. Color change measurements ( $\Delta E$ ) for four different  $TiO_2/PdO$  pigments after 2 min of exposure time to 1%  $H_2$  gas.

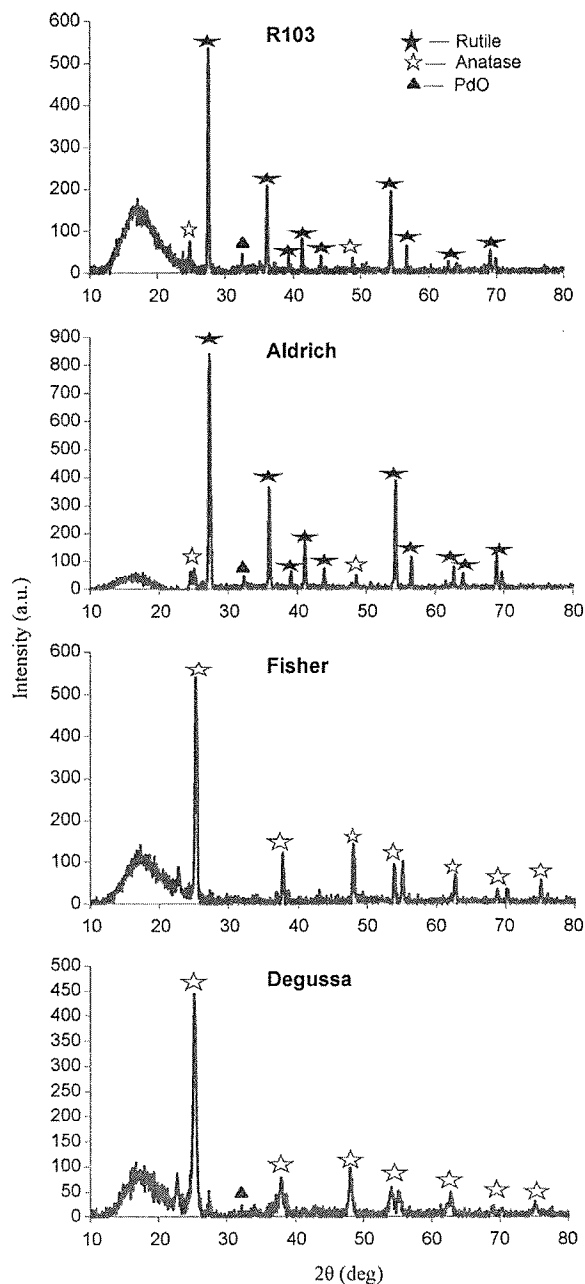


Fig. 3. XRD spectra of  $TiO_2/PdO$  pigments.

### 3.3. X-ray photoelectron spectroscopy

To better understand the chemistry behind the pigment color transition and response to environmental effects, XPS data was collected on the Aldrich  $TiO_2$  pigment prior to any hydrogen exposure. Fig. 4 shows the wide survey scan of the pigment. It contains Pd, Ti, O, Na, C, and Cl.

Fig. 5 shows the high resolution spectra of the Pd and Ti peaks. The Pd  $3d_{5/2}$  peak was measured at 337.5 eV, corresponding to PdO in agreement with the literature [10–13]. The peak position for  $Pd(OH)_4$  has been reported at 338.5 eV [12]. From the spectra in Fig. 5a, the peak at 337.5 eV dominates; therefore, presence of hydroxide is in all likelihood negligible.

Fig. 5(b) shows distinctively that the Ti is present as  $TiO_2$  (459.8 eV) [10]. No shoulders were observed on the main peak that

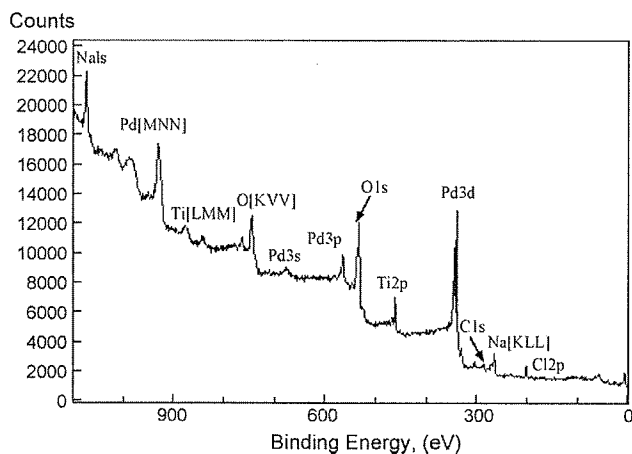


Fig. 4. XPS wide survey scan indicating the presence of Pd, Ti, O, Na, C, and Cl in the pigment.

could be fitted with sub-oxides or other species. The oxygen spectrum (not shown) was complicated by the presence of the large Pd 3p peak, and thus positive identification was not possible.

Fig. 6 shows spectral overlays for pure palladium metal, the pigment unexposed to H<sub>2</sub> gas, and pigment exposed to H<sub>2</sub> gas. The C 1s peak is shown as a reference, 284.6 eV and it occurs at the same binding energy for all three specimens. The dashed vertical line is aligned with the peak for the Pd metal. The peak for

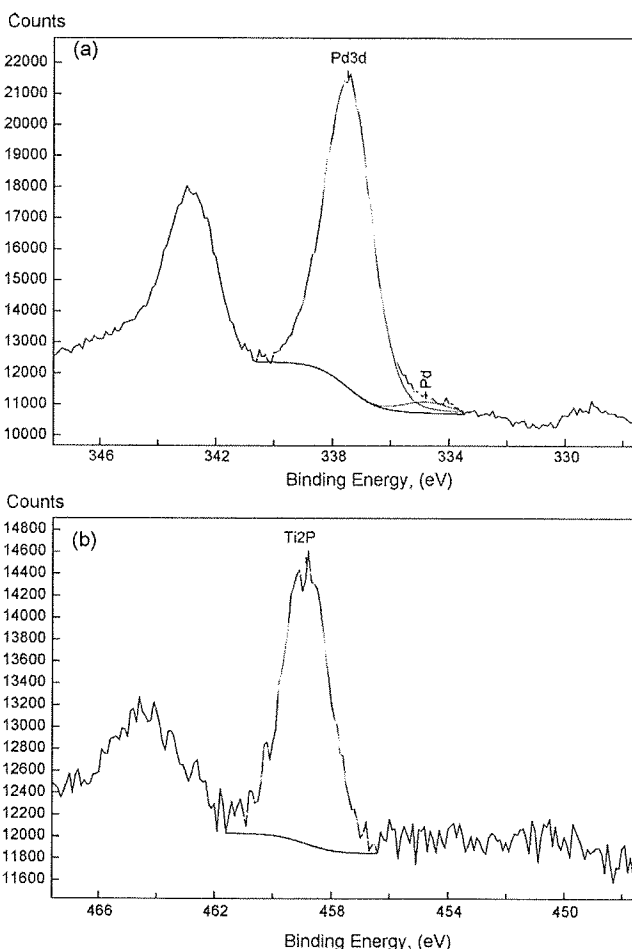


Fig. 5. XPS high resolution spectra of the Pd (a) and Ti (b) peaks in the pigment.

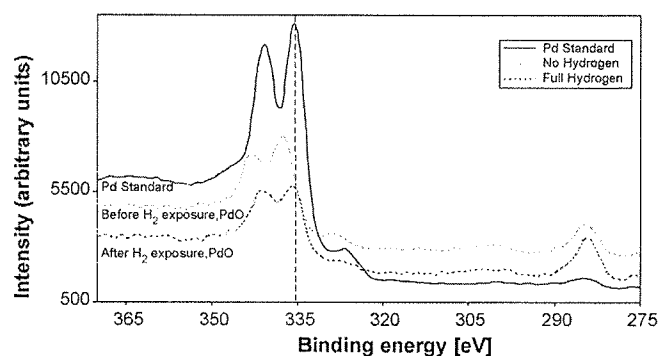


Fig. 6. XPS spectra of the Pd peak for pure Pd metal, unexposed pigment, and pigment exposed to H<sub>2</sub> gas.

PdO in the unexposed pigment is shifted to the left of the vertical line. However, the binding energy for the pigment exposed to the H<sub>2</sub> gas lines up with the peak for the pure palladium metal. As mentioned before, when the pigment is exposed to H<sub>2</sub> gas, PdO is reduced to elemental Pd metal and the binding energy shifts to the right.

### 3.4. PdO/TiO<sub>2</sub> particle characterization

To obtain information on the size and morphology of the pigments and to explore the underlying chemistry and role of the TiO<sub>2</sub> support in the coloration reaction, the pigments were subjected to a detailed Transmission Electron Microscopic study [7,8].

Fig. 7 shows TEM images of various PdO/TiO<sub>2</sub> pigments using different titania supports. Although all pigments had been synthesized in the same manner, nonetheless it can be seen that the size of PdO particles and distribution vary considerably. PdO particles are clearly visible as dark colored areas on the surface of the TiO<sub>2</sub> particles. In the case of Degussa P-25 and Aldrich TiO<sub>2</sub>, the hemispherical PdO crystallites were well dispersed and strongly attached to the metal oxide support at their flat planes (Fig. 7A–D) [7].

The faster response time of Degussa P-25 based pigments compared to that of Aldrich could be explained in terms of the size and homogeneity of PdO particles and their interaction with TiO<sub>2</sub> surfaces. The PdO particle size for Degussa P-25 was homogenous with an estimated mean particle diameter of 2.0 nm. On the other hand, the size of the PdO particles on the Aldrich TiO<sub>2</sub> was less homogenous and varied from 2 nm to 10 nm, with an estimated log mean particle size of 3.4 nm.

Fisher and DuPont R103 supported pigments (Fig. 7E–H) gave similar results when exposed to hydrogen gas. The mean diameter of PdO particles for Fisher and DuPont R103 pigments 3.1 nm and 2.5 nm, respectively. In both cases, although the particle size distribution were similar to Degussa P25 but their shapes were more spherical and, as a result had less interaction with the TiO<sub>2</sub> support surface as compared to Degussa P-25 and Aldrich supported pigments.

Clearly, from the TEM images, it is clear that PdO did not wet the titania. Qualitatively, it appeared that the Aldrich titania caused the PdO particles to be larger and somewhat more agglomerated. To quantify this observation, particle diameters were measured one at a time manually using ImageJ [14]. The particle diameters were then grouped into logarithmically evenly spaced bins ranging from 1 nm to 100 nm in diameter, with 10 bins per factor of 10 change in dimension. Agglomeration is easiest to quantify from the geometric standard deviation,  $\sigma$ , as defined in the cumulative log-normal distribution in Eq. (3) [15–18]. This can be achieved using the NORMINV function in Excel using a probit mean of five and a

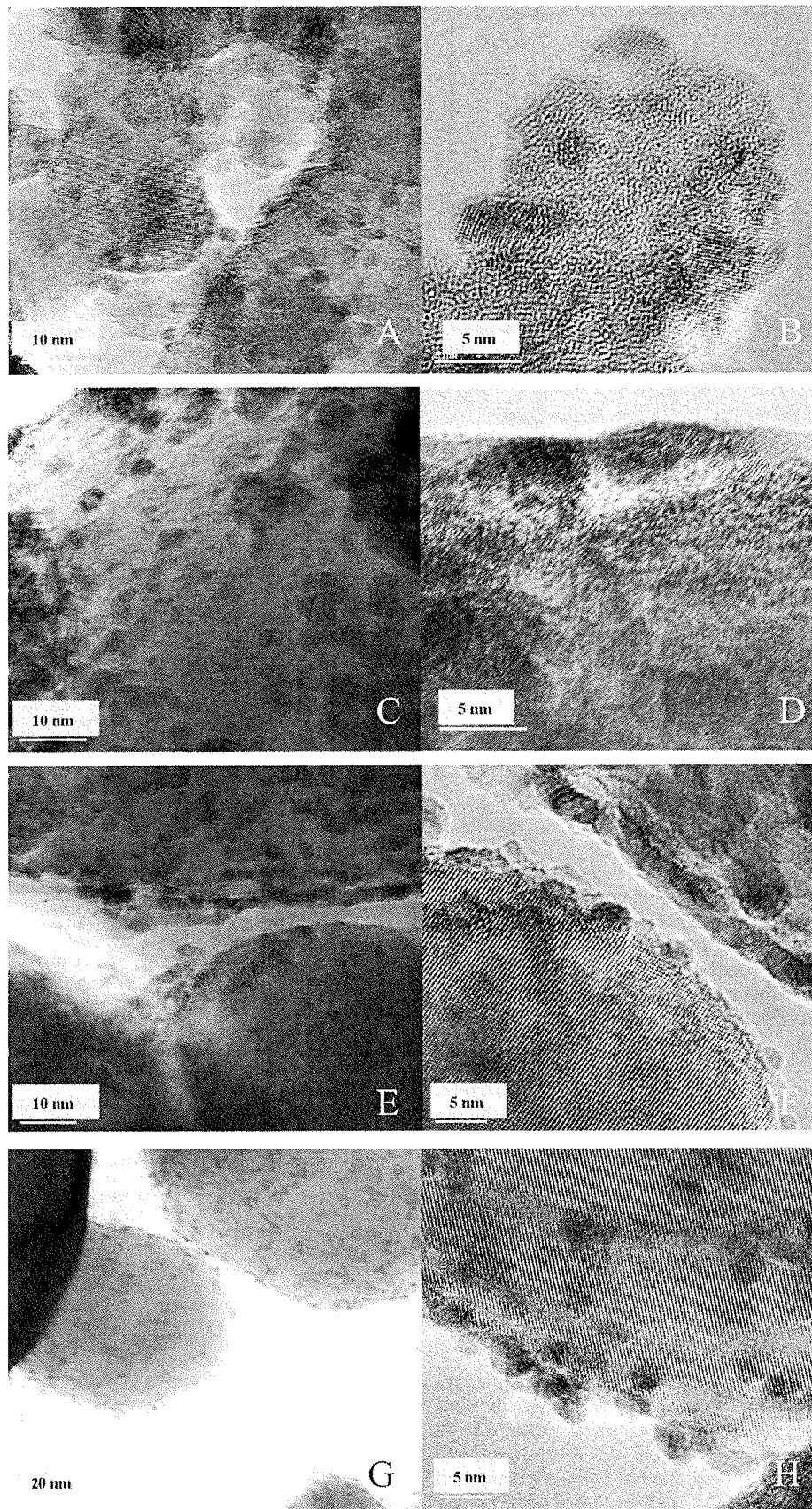


Fig. 7. TEM images of (A and B) PdO/Degussa P-25 TiO<sub>2</sub>, (C and D) PdO/Aldrich TiO<sub>2</sub>, (E and F) PdO/Fisher TiO<sub>2</sub>, and (G and H) PdO/DuPont R103 TiO<sub>2</sub> pigments.

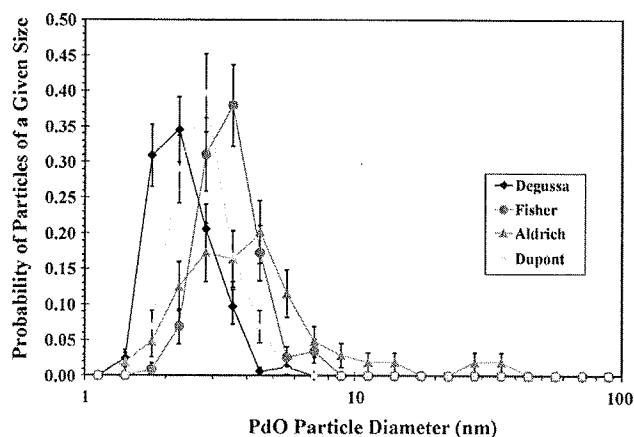


Fig. 8. PdO particle size distribution on various titania support.

Table 1  
Effect of titania vendor on PdO particle size.

Titania vendor	Log mean diameter (nm)	Error in log mean diameter (nm)	G.S.D.	Error in G.S.D.
Degussa	2.0	0.2	1.3	0.1
Dupont	2.5	0.3	1.3	0.2
Fisher	3.1	0.4	1.3	0.1
Aldrich	3.4	0.4	1.6	0.2

probit standard deviation of unity.

$$F_{LN}(d) = 0.5^* \left[ 1 + \operatorname{erf} \left[ \frac{\ln(d_i/\bar{d})}{2^{0.5} \ln(\sigma)} \right] \right] \quad (3)$$

In Eq. (3),  $d$  is a given particle diameter,  $F_{LN}(d)$  is the cumulative lognormal probability density function,  $\bar{d}$  is the log mean diameter and  $\sigma$  is the geometric standard deviation. From Eq. (3), one can show that the geometric standard deviation is equal to the diameter corresponding to a cumulative probability of 0.8413 divided by the log mean diameter. Granqvist and Buhrman [15] have shown that Eq. (3) can be derived from particle coalescence theory, which is valid for all but the narrowest particle size distributions ( $\sigma < 1.2$ ). The PdO particle size distributions as a function of titania vendor are summarized in Fig. 8.

The log mean particle diameters and geometric standard deviations are summarized in Table 1. The difference in particle diameter between the Degussa and Dupont titania is barely statistically significant at the 95% confidence level, and the same is true between the Dupont and the Fisher titanias. The one overwhelming difference is the breadth of the distribution of PdO particles on the Aldrich titania. One can clearly see from the geometric standard deviation (GSD) results that the distribution is far broader for the Aldrich titania than for any of the others. PdO agglomeration is only significant for the Aldrich titania.

### 3.5. Environmental exposure test

Many of the various environmental and laboratory exposures did affect the extent of color change in the chemochromic samples when exposed to  $H_2$  gas. When the tape was exposed to DI water, salt water, UV light, and heat, the extent of color change was more pronounced. At the KSC beach site, the tapes were exposed to salt water, heat, and UV light. The combination of these factors caused the tape at the beach site to change color faster than the one tested in the laboratory environment. The chemistry underlying this accelerated color change is currently under investigation.

## 4. Conclusions

Four different chemochromic  $TiO_2$ -supported PdO pigments were synthesized, and their activity toward hydrogen was measured. Degussa P-25 based pigments yielded the fastest discoloration kinetics toward  $H_2$  gas, while the Aldrich titania (mainly rutile crystalline form) based pigments had the greatest  $\Delta E$ , color contrast, value. The chemochromic activity of the pigments toward hydrogen is a strong function of the particle size and level of PdO agglomeration on the  $TiO_2$  support surface. Smaller PdO particles with a hemispherical crystalline structure produced faster kinetics when exposed to  $H_2$  gas. However, agglomeration of PdO particles on the surface of  $TiO_2$  gave larger color contrast number,  $\Delta E$ . Samples with spherical particles resulted slower kinetics as well as smaller color change. XRD analyses show that  $TiO_2$  crystalline phase apparently had no effect on the chemochromic performance of these pigments in laboratory environment.

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## Biographies

Nahid Mohajeri is a Research Chemist at Florida Solar Energy Center since 2002. She holds a doctorate in chemistry from Florida Institute of Technology. Her main areas

of research interest include: thermal stability of polymers and electrochemistry, hydrogen storage, and hydrogen sensor. She is the author or co-author of more than one dozen scientific publications on topics ranging from synthetic organic chemistry, to thermal stability of polymers and metal hydrides for hydrogen storage. Her current research is in proton exchange membranes for fuel cell applications.

**Ali T-Raissi** is the Director of FSEC's Hydrogen R&D Division and a 25-year veteran of hydrogen R&D. He holds a PhD in mechanical engineering from the University of California at Berkeley. He is author of more than 140 professional papers and technical reports related to hydrogen production, storage and detection technologies, and photocatalysis funded by the US DOE, US Navy (IHDNSWC), ONR (AASERT), Army (CERL), ARO (DURIP), US EPA (GCHSRC), NASA-KSC, NASA-GRC, DuPont Corp., Radian Corp., TAPPI Foundation, and others.

**Gary Bokerman** holds a PhD in chemistry from the University of Michigan and has retired from Dow Corning Corporation, a specialty chemical manufacturer focused on silicon technology, in 2000. During part of his 32-year industrial career, he conducted and directed research on basic processes to produce organosilicon compounds. In 2004 he joined the Hydrogen Division of the Florida Solar Energy Center as a winter time researcher. His research areas include rheological behaviors of liquid hydrogen tank insulation, chemochromic hydrogen sensor matrices, and improved methods to chemically produce hydrogen for fuel cells.

**Janine E. Captain** received a BS in chemistry and Marine Science from the University of Miami and a PhD in chemistry from Georgia Institute of Technology. Her graduate work focused on surface chemistry on icy surfaces related to atmospheric chemistry. Janine has been working for NASA at the Kennedy Space Center since 2005 focusing on in situ Resource Utilization technologies and sensors for field deployment. Her work on sensors includes detection of hydrogen and hypergolic fuels.

**Barbara V. Peterson** is an analytical chemist experienced in environmental chemistry, and technology development. This includes work with The Environmental Protection Agency, Battelle-PNL, Dow Chemical and Kennedy Space Center, KSC

contractors. Her contributions to EPA publication, *Measuring and Estimating the Bio-concentration Factor of Chemicals in Fish* received Chem Abstracts Classical Citation Status and is a basis for environmental legislation. At KSC, she developed the analytical laboratory for NASA's Controlled Environment Life Support program for long term space flight and colonization. She coordinated water quality studies to define ecological management requirements. She worked in the Technology Transfer office and with sensor development.

**Mary Whitten** earned her PhD from Northern Illinois University where her research involved in situ phosphatizing coatings. Upon graduation, she took a position as an Assistant Professor of Chemistry at the University of the Virgin Islands. Currently, she is a subcontractor at the Kennedy Space Center through the University of Central Florida working on various projects which aid ground support equipment at the launch pads.

**Steve Trigwell** received the BSc degree in physics from Leicester University, UK, the MS degree in materials engineering from San Jose State University, CA, and the PhD degree in applied science from the University of Arkansas at Little Rock, AR. He is currently the Manager of the Applied Science and Technology Department at ASRC Aerospace Corporation at Kennedy Space Center. His group is currently involved in all areas of lunar exploration for NASA's commitment to returning to the Moon. He is a Fellow of the Institute of Materials, Minerals, and Mining in the UK.

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**James Brenner** joined the chemical engineering faculty at Florida Tech in 1998 and since then, he has worked in H<sub>2</sub> storage, purification, and sensing using metal hydride thin films on porous stainless steel (PSS) and templated porous carbon (TPC)/PSS composite supports. Dr. Brenner has performed research projects on bio-diagnostic chips, electronic noses, metallic nanoparticles, polymer foams for the space shuttle's external tanks, pillared molybdenum sulfide, nitride, and carbide catalysts, and characterization of and catalyst development for upgrading of coal, heavy oil, and lignin feedstocks.

