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Changes in the Optical Properties of Simulated Shuttle Wastewater Deposits: Urine Darkening

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Manned spacecraft have historically dumped the crew-generated wastewater overboard, into the environment in which the spacecraft operates, sometimes depositing the wastewater on the external spacecraft surfaces. The change in optical properties of wastewater deposited on spacecraft external surfaces, from exposure to space environmental effects, is not well understood. Nonvolatile residue (NVR) from human urine is used to simulate wastewater deposits and document the changes in the optical properties of the NVR deposits after exposure to ultraviolet (UV) radiation. There are 20 NVR samples, from 0 to 1000 Å/cm^2, and one sample contaminated with 1–2 mg/cm^2, exposed to UV radiation over the course of approximately 6151 equivalent sun hours. Random changes in sample mass, NVR, solar absorbance, and infrared emission were observed during the study. Significant changes in the UV transmittance were observed for the sample contaminated at the milligram per square centimeter level.

Introduction

THE Mir Environment Exposure Payload, which included flight experiments such as the Polished Plate Meteoroid Detector (PPMD) and the Passive Optical Sample Assembly II (POSA-II), was delivered to the Russian space station Mir aboard space transportation system (STS)-76 and returned to Earth aboard STS-86. The experiments, mounted in passive experiment containers (PECs), were exposed to the Mir external environment for 18 months, during which time four space shuttle flights approached and docked with Mir. Postflight inspection of the external PPMD and POSA-II surfaces revealed material that, from the pattern of the nonvolatile residue (NVR) deposits, appeared to have been a liquid when the material contacted the surfaces of both the PECs and the experiments.

Chemical analysis of the deposited material detected inorganic salts that are commonly found in human urine, which suggested that wastewater released into the external Mir environment was the source of the contaminant. To document and quantify the effect of ultraviolet (UV) radiation on simulated wastewater, a study was conducted as a joint effort between NASA Marshall Space Flight Center (MSFC) and the NASA Johnson Space Center (JSC). The study tracked the changes in the trasmittance, from 200 to 2500 nm, of urine deposits after multiple UV exposure intervals totaling approximately 6151 equivalent sun hours (ESH) of UV exposure.

Method

Fused silica disks, 2.54 cm in diameter, were used as the substrate on which the simulated wastewater NVR was deposited. This material was selected based on its optical transmittance, 170–2500 nm, and the chemical inertness of the silica (Fig. 1).

To increase the amount of sample solution that could be applied to a substrate in a single application, and to ensure uniform wetting of the entire substrate surface, a wall or dike was formed around the edge of each substrate using Kapton® tape (acrylic adhesive). This modification allowed 1.0 ml of the sample solution, containing the desired amount of material, to wet the entire substrate surface uniformly. The tape was then removed from the substrate after the solution had evaporated. The same Kapton tape was applied to the surface of two substrates, samples 19 and 20, before the application of the urine solution. This layer of Kapton was intended to be a low-fidelity simulation of the Kapton substrate of the International Space Station solar arrays. The procedure for the application of the urine solution to these samples was the same as all of the other samples.

Simulated Wastewater/Urine

The urine used in the study is commonly used as an analytical standard by medical laboratories and was purchased from Bio-Rad Laboratories, Hercules, California, as a dehydrated material, Lyphocheck® (a lyophilized human urine-based quality control product). A density of 1.0 g/cm^3 was assumed for the material, which was rehydrated with 18-MΩ water. A stock solution of 5.1×10^{-5} g/ml (1000 Å/ml) was prepared, and serial dilutions of this solution made. The concentration of urine, in the serial dilutions, was calculated to produce the desired NVR level (in angstroms per square centimeter) when 1.0 ml of the solution was placed on the substrates. The sample solutions were prepared immediately before application of the solution to the fused silica substrate and, once applied to the substrates, allowed to air dry in a laminar flow bench that provided a clean environment for the drying process.

Preliminary optical and mass measurements were made on all 24 samples to document the initial properties of the samples before UV exposure. A review of the measurements did not indicate any significant differences between clean substrates and those with NVR deposits (Fig. 2). Additional ellisipsometery measurements also failed to detect material on the substrates.

Because measurable differences in the samples could not be demonstrated, two grossly contaminated samples were prepared by JSC to ensure that the study did contain a sample for which the initial optical properties could be measured. The same methods used to create the initial 24 samples were used to create two samples contaminated at the milligram per square centimeter level. One of the substrates was a 2.54-cm-diam zinc selenide disk, which was used to confirm the presence of the urine deposit on the substrate by Fourier transform infrared spectroscopy (FTIR). The other substrate was a fused silica disk identical to, and purchased with, the substrates used to create the original 24 samples. This sample was labeled JSC-B,

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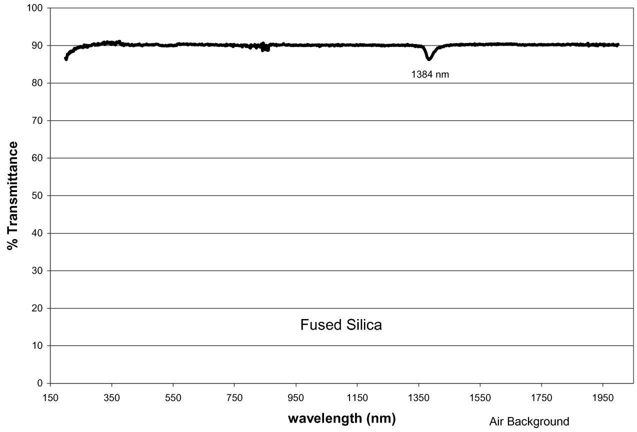

Fig. 1 UV/VIS spectrometer scan of the fused silica substrate 200–2000 nm; ambient laboratory air used as scan background.

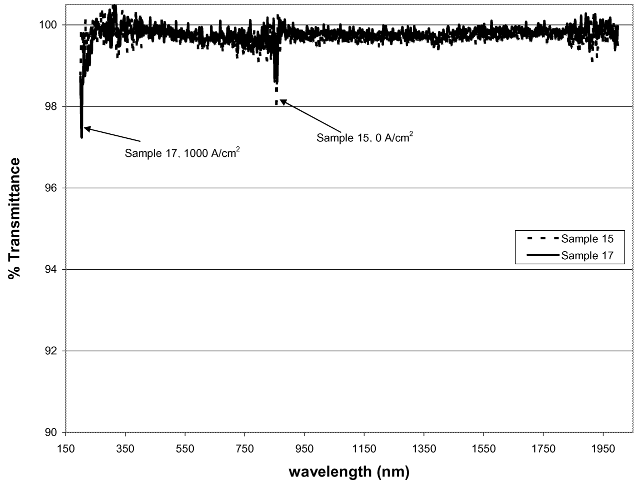

Fig. 2 Samples 15 and 17 before UV exposure.

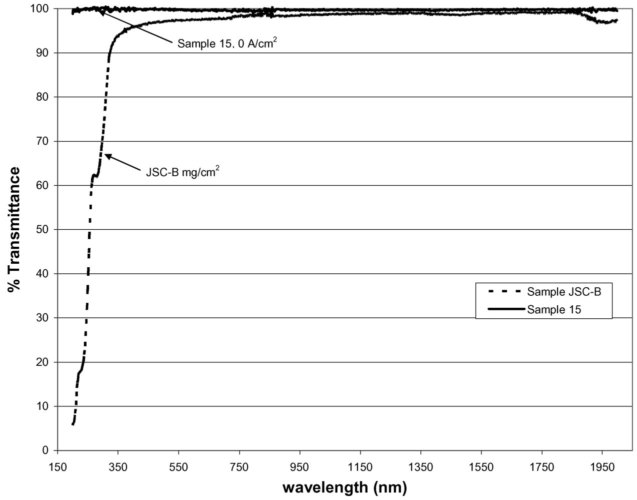

Fig. 3 UV/VIS scan of sample JSC-B before UV exposure.

and the NVR deposit could be seen on the surface of the substrate. The presence of the NVR was also confirmed spectroscopically by the UV/visible radiation (UV/VIS) scan (200–2000 nm) of this sample (Fig. 3). Four of the original 24 substrates, two with 1000 Å/cm^2 and two with 0 Å/cm^2 of the simulated urine deposit, were set aside as controls and would receive no UV exposure.

MSFC UV Exposure Facility

The vacuum chamber, in which the exposure of the samples to UV radiation was carried out, was assembled by MSFC for this study. The vacuum chamber is evacuated by a 470-liter/s turobomolecular pump backed by a direct drive roughing pump capable of evacuating the chamber to a hard vacuum ($<10^{-6}$ torr). The chamber is constructed of stainless steel and has a 19.5-cm-diam, UV-transparent, viewing port through which the samples were illuminated.

The samples were mounted on an aluminum sample holder (Fig. 4) that was bolted to an actively cooled fixture in the vacuum chamber. The samples were maintained at 20°C during the

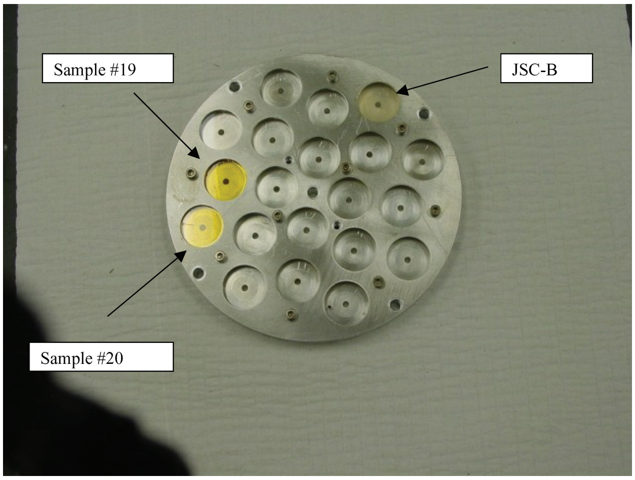

Fig. 4 Sample holder with fused silica sample disks as they appeared after approximately 6151 ESH of UV exposure; on the left-hand side of the sample holder (9 and 10 o'clock positions) are samples 19 and 20; dark sample at the upper right-hand side of the sample holder (1 o'clock) is sample JSC-B.

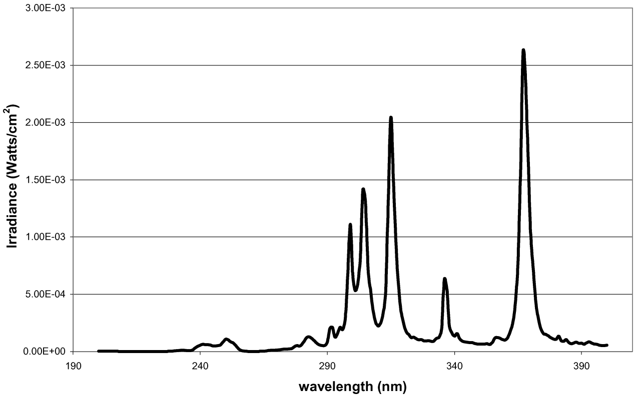

Fig. 5 UV lamp spectrum, major peaks at 304, 315, and 376 nm.

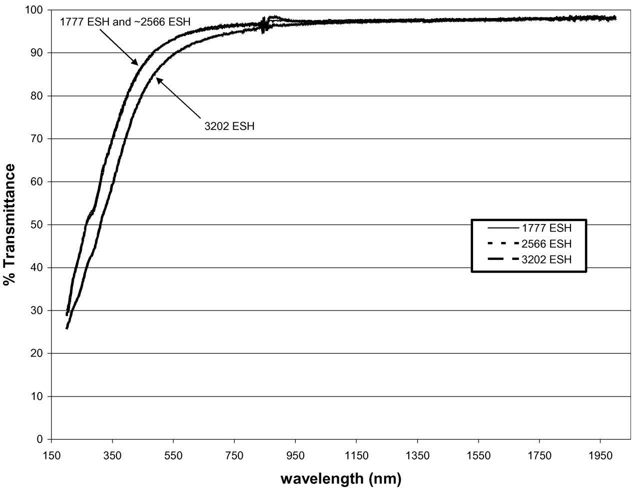

Fig. 6 Scans of JSC-B before and after UV/VIS lamp failure.

exposure periods by the circulation of chilled water through the mounting fixture

The samples were illuminated with a mercury xenon lamp (200–2500 nm) positioned 74 cm away from the outer surface of the UV-transparent chamber viewing port. During the exposure periods, the intensity of the UV at the surface of the viewing port was checked with a hand-held radiometer. The silicon, photodiode radiometer has a flat response filter, combined with a wide-eye diffuser, that provides a flat spectral response from 410 to 1000 nm. With the vacuum chamber at ambient pressure (laboratory atmosphere), the measured incident light at the sample holder, 2.2 mW/cm^2, was 61.1% of the light intensity measured at the surface of the viewing port. The sample holder is located 27 cm behind the outer surface of the viewing port.

UV Lamp Failure

During the exposure period of 1775–2566 ESH, the UV/VIS lamp failed, terminating the illumination of the samples. No rationale for the failure, such as a facility power failure, was identified, and the lamp functioned normally when restarted. Before the lamp failure, the output from the UV/VIS lamp was measured at the beginning and conclusion of each exposure period (Fig. 5). The frequency of these measurements was increased to a daily measurement to ensure that another lamp failure would be detected before the end of an exposure period. Based on the measure change in UV transmittance of the sample JSC-B (Fig. 6), it is believed that the lamp failure occurred early in this exposure period. The sample exposure was extended beyond 4000 ESH to compensate for the lamp failure, and the total exposure time, 6151 ESH, could be reduced by as much as 789 ESH.

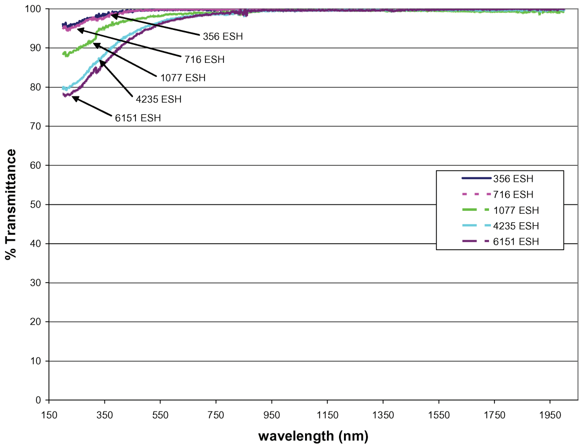

Fig. 7 Progressive darkening of sample 15 at various, intermediate exposures.

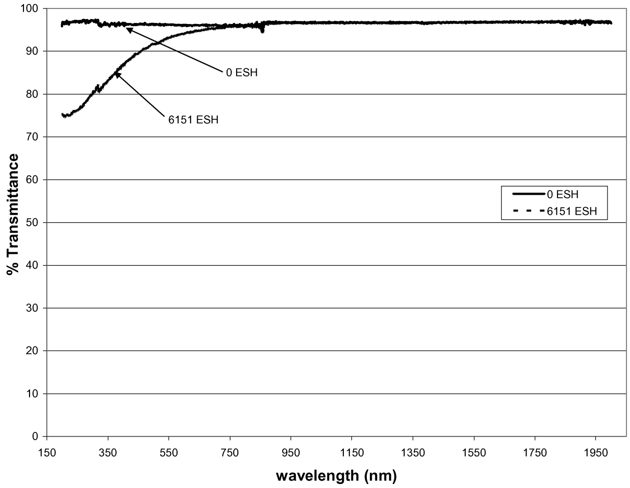

Fig. 8 Sample 15, 0 Å/cm^2, showing loss of transmittance or substrate darkening.

Discontinued Measurements

In addition to UV/VIS transmittance measurements, the original study protocol included the weighing of the samples, measurement of the solar absorbance (200 and 2800 nm), and the measurement of the infrared emittance (2000–20,000 nm) of the samples after each exposure period. After the first three exposure periods of 100, 358, and 716 ESH, only random fluctuations were recorded for these parameters, and no significant change from the preexposure values were observed. After the measurements at 716 ESH, no future mass, solar absorbance, or infrared emittance measurements were made

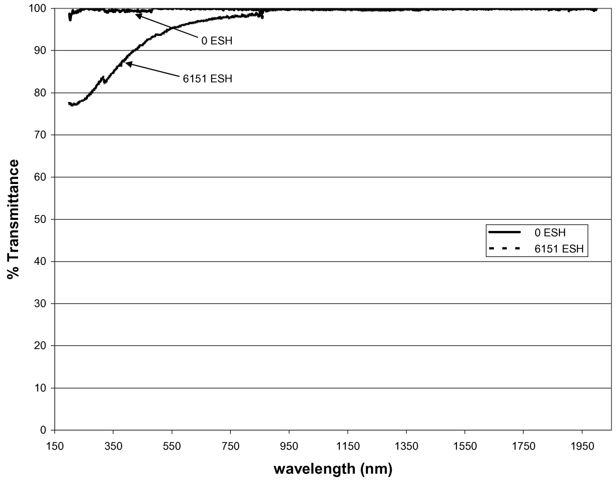

Fig. 9 Sample 17, 1000 Å/cm^2, also showing loss of transmittance or substrate darkening.

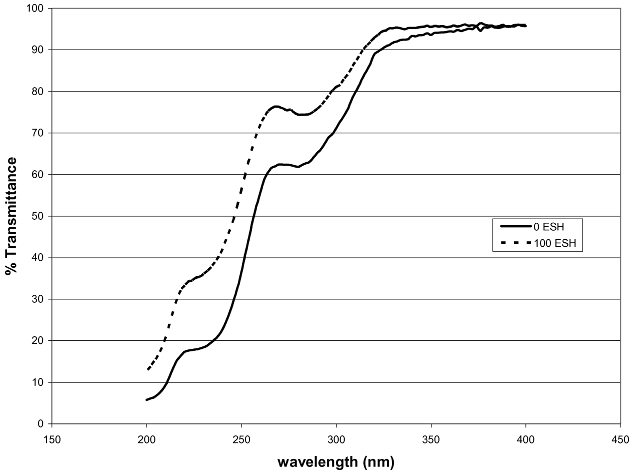

Fig. 10 Sample JSC-B showing an increase in transmittance after the initial exposure to vacuum conditions and 100 ESH of UV/VIS.

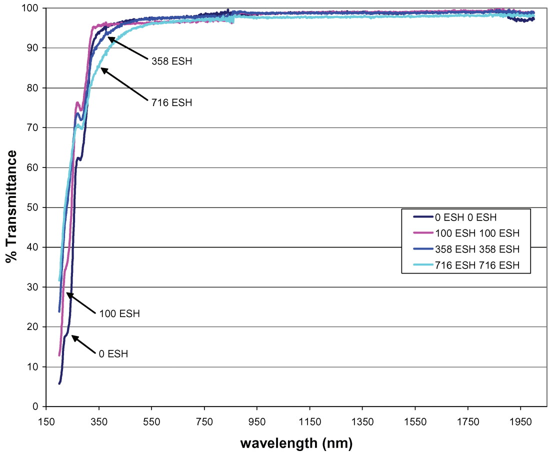

Fig. 11 UV/VIS spectra of sample JSC-B from 0 to 716 ESH.

until after the final UV exposure. After approximately 6151 ESH of UV exposure, no significant changes in these parameters were measured.

UV/VIS Spectrometer Measurements

A Perkin-Elmer Lambda-19 UV/VIS spectrometer was used to measure the changes in transmittance of the samples over the spectral range from 200 to 2500 nm. The preexposure measurements, transmittance and reflectance, failed to show any difference between the spectrum of the bare substrates and those with the NVR deposits (Fig. 2). Sample JSC-B did have a unique transmittance spectrum, which was attributed to the substantial NVR deposit on the substrate.

General Discussion

Substrate Darkening

The transmittance of the UV-exposed substrates was observed to decrease over the wavelength range from 200 to 860 nm with each additional UV exposure. The final UV transmittance was about 75% of the original transmittance, and the decrease in transmittance appeared to be independent of amount of NVR believed to be deposited on the substrate, as shown in Figs. 7–9.

Sample JSC-B

The grossly contaminated sample prepared by JSC, an addition to the original 24 samples, did have strong absorbance bands in the region from 200 to 320 nm, before any UV exposure. The transmittance of the sample increased during the first 100 ESH of UV exposure (Fig. 10). This increase in transmittance was attributed to a loss of material from the surface of the substrate, possibly outgassing, and was not observed in subsequent UV exposure periods. With increased UV exposure, the transmittance of the sample, in the wavelength range of 200–650 nm, continued to decrease, and the absorbance bands merged into a single, broad curve after 1077 ESH, as shown in Figs. 11–16.

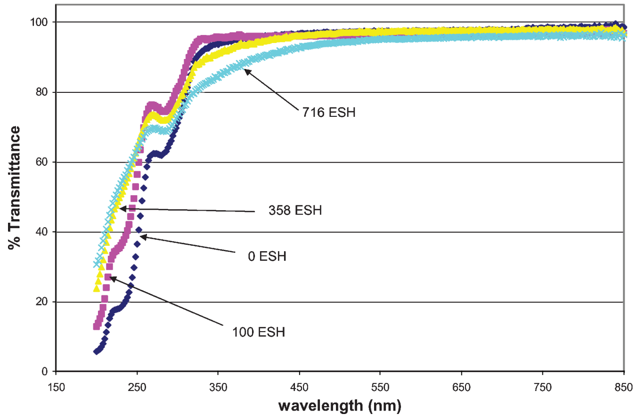

Fig. 12 **UV/VIS spectra of sample JSC-B from 200 to 850 nm for the exposure period of 0–716 ESH.**

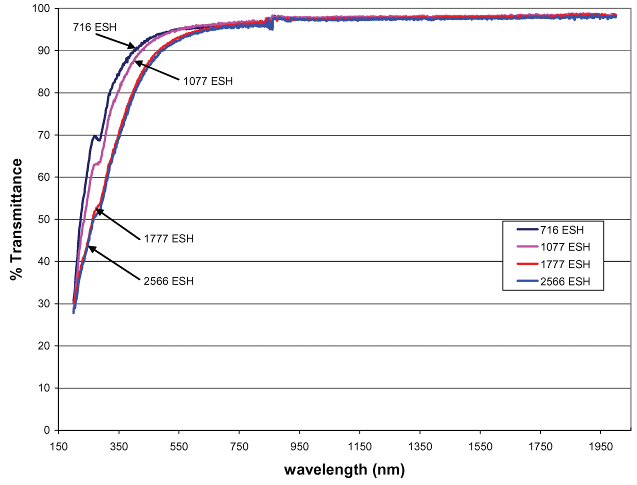

Fig. 13 **UV/VIS spectra of sample JSC-B from 716 to 2566 ESH.**

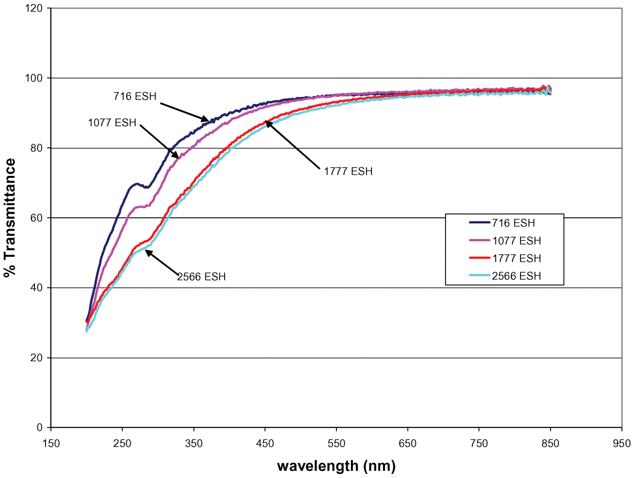

Fig. 14 **UV/VIS spectra of sample JSC-B from 200 to 850 nm for the exposure period of 716–2566 ESH.**

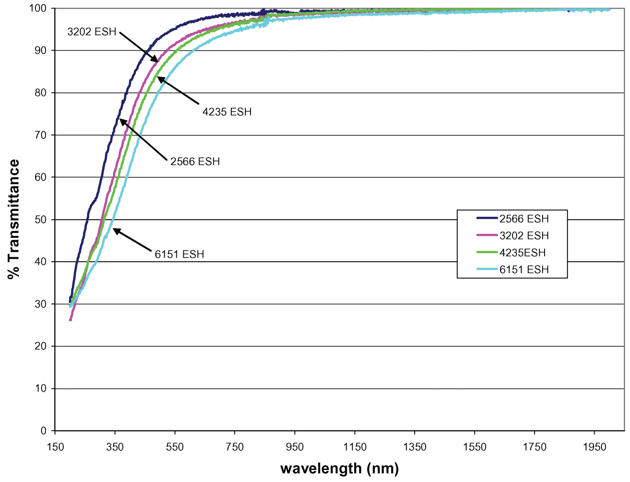

Fig. 15 **UV/VIS spectra of sample JSC-B from 2566 to 6151 ESH.**

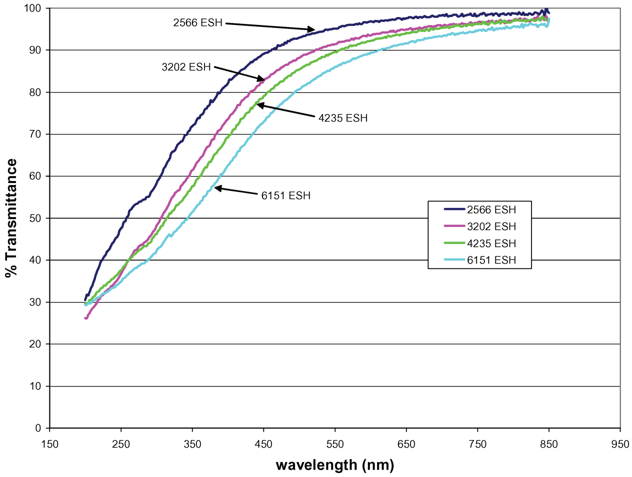

Fig. 16 **UV/VIS spectra of sample JSC-B from 200 to 850 nm for the exposure period of 2566–6151 ESH.**

Kapton Samples

Two of the samples, 19 and 20, differed from the other samples because Kapton tape had been applied to the fused silica substrates

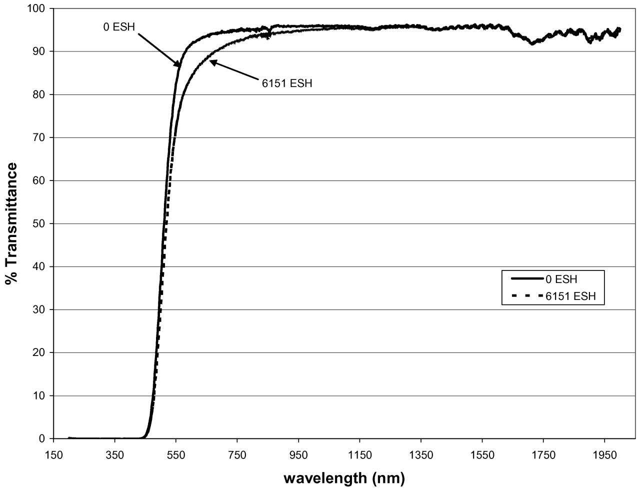

Fig. 17 UV/VIS spectrum of sample 19.

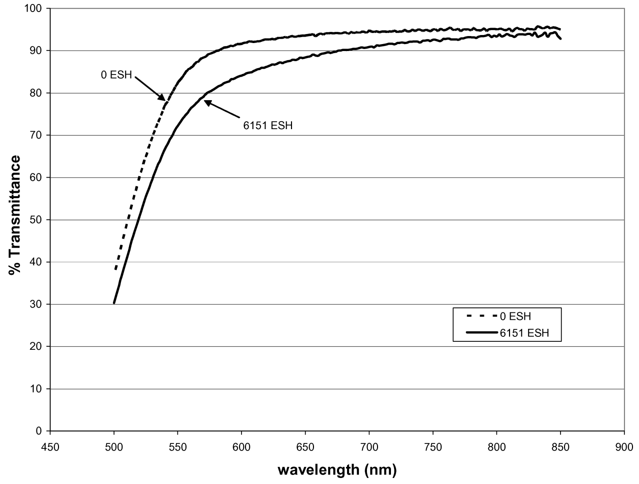

Fig. 18 Section of interest from the composite curve of sample 19.

before the urine solution was applied to the composite substrate. The target NVR deposit on these samples was 500 Å/cm^2, similar to the NVR deposits on samples 13 and 14. These composite samples have a broad absorbance band from 200 to 650 nm, which is attributed to the Kapton tape (Figs. 17 and 18).

Conclusions

A measurable decrease, maximized at 200 nm, of approximately 25% in optical transmittance, was observed over the wavelength range of 200–900 nm for the fused silica substrates used in this study as the exposure to the UV source progressed. No significant changes in the solar absorbance, infrared emittance, sample mass, or transmittance (200–2500 nm) were measured for urine deposits of 1000 Å or less. A single urine deposit at the milligram per square centimeter level did undergo a measurable decrease in transmittance as the UV exposure progressed.

Spectra of urine deposited on Kapton, applied over the fused silica substrates used in this study, did show a small change in transmittance (200–2500 nm). Similar urine deposits placed directly on the fused silica substrates did not undergo the same change in transmittance, suggesting that the change can be attributed to the Kapton layer.

The darkening of the urine deposits, as measure spectrographically, may be affected by the morphology or the concentration of the urine deposits. It was assumed that the urine deposits would form a semi-uniform film across the surface of the substrates. Elipsometer measurements of the urine deposits were inconclusive for deposits of 1000 Å or less, and no attempt to characterize the morphology of the milligram per square centimeter sample was made. It is possible, but not demonstrated in this study, that a threshold may exist for either the detection of the urine on the fused silica substrates or the UV-induced change in transmittance.

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