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Technical note / Note technique

Characterization of a low-cost diffuse reflectance coating

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Abstract. A simply formulated, inexpensive, and highly reflective diffuse coating was characterized with respect to its reflectance, bidirectional reflectance factor (BRF), minimum thickness, application method, abrasion, and response to weathering. The average reflectance of the coating between 400 and 1600 nm was 94.9 ± 0.13% (minimum reflectance of 86.4 ± 0.28% at 400 nm, maximum reflectance of 98.3 ± 0.14% at 989 nm) when spray-applied, which was slightly lower than, but still comparable to, that of commercially available coatings. The application method was shown to affect reflectance, BRF, and the effects of abrasion on BRF. Overall, spray application gave the best combination of high reflectance and close-to-ideal diffusion profile across the 400–1600 nm range. The coating became more specular with increased abrasion, regardless of application method, but the effect was most prominent with brush-applied coatings at longer wavelengths. The impact was slight enough, given the stiff brush used in testing, that infrequent light brushing with a soft-bristled brush would not adversely affect the coating properties. Weathering resulted in a decrease in reflectance of approximately 2%, with an increase in reflectance variability among samples and increased brittleness.

Résumé. Un revêtement à surface de rayonnement diffus très réfléchissant, de formulation simple et de faible coût, a été caractérisé en termes de sa réflectance, du facteur de réflectance bidirectionnelle (FRB), d'épaisseur minimale, de méthode d'application, d'abrasion et de réponse aux effets d'exposition aux intempéries. La réflectance moyenne de la couche de revêtement était de 94,9 ± 0,13 % entre 400 et 1600 nm (réflectance minimale de 86,4 ± 0,28 % à 400 nm, et maximale de 98,3 ± 0,14 % à 989 nm) lorsque appliquée par pulvérisateur, ce qui était légèrement plus faible mais comparable aux revêtements disponibles sur le marché. Il a été démontré que le mode d'application affecte la réflectance, la FRB et les effets d'abrasion sur la FRB. Globalement, l'application par pulvérisateur a donné la meilleure combinaison de réflectance élevée et de profil de diffusion quasi idéal à travers l'intervalle de 400–1600 nm. Le revêtement est devenu plus spéculaire avec l'accroissement de l'abrasion, indépendamment du mode d'application, mais l'effet était plus visible avec les revêtements appliqués au pinceau à des longueurs d'onde plus longues. L'impact était suffisamment faible, compte tenu du pinceau plutôt rigide utilisé durant les tests, qu'une application superficielle occasionnelle avec un pinceau à soies souples ne devrait pas affecter de façon négative les propriétés du revêtement. L'exposition aux intempéries a résulté en une réduction de la réflectance d'environ 2 %, avec un accroissement de la variabilité au niveau de la réflectance entre les échantillons et une augmentation de la friabilité.
[Traduit par la Rédaction]

Introduction

Diffuse reflectance materials are characterized by their nonspectral reflectance; they reflect light uniformly in all directions. The ideal diffuse reflector is a Lambertian surface, being totally reflective (having 100% reflectance), and radiance being independent of direction (Schaepman-Strub et al., 2006). Whereas no perfectly Lambertian surfaces exist that are practical for laboratory and (or) field use, materials have been created that are close to ideal.

One important application of diffuse reflectance is in integrating spheres (**Figure 1**). Integrating spheres combine the uniform reflectance of a Lambertian surface and the spherical geometry "to spatially integrate radiant flux" (Labsphere Inc., 1998). Integrating spheres are used to provide a uniform light source, to measure the flux of lamps, and to measure total

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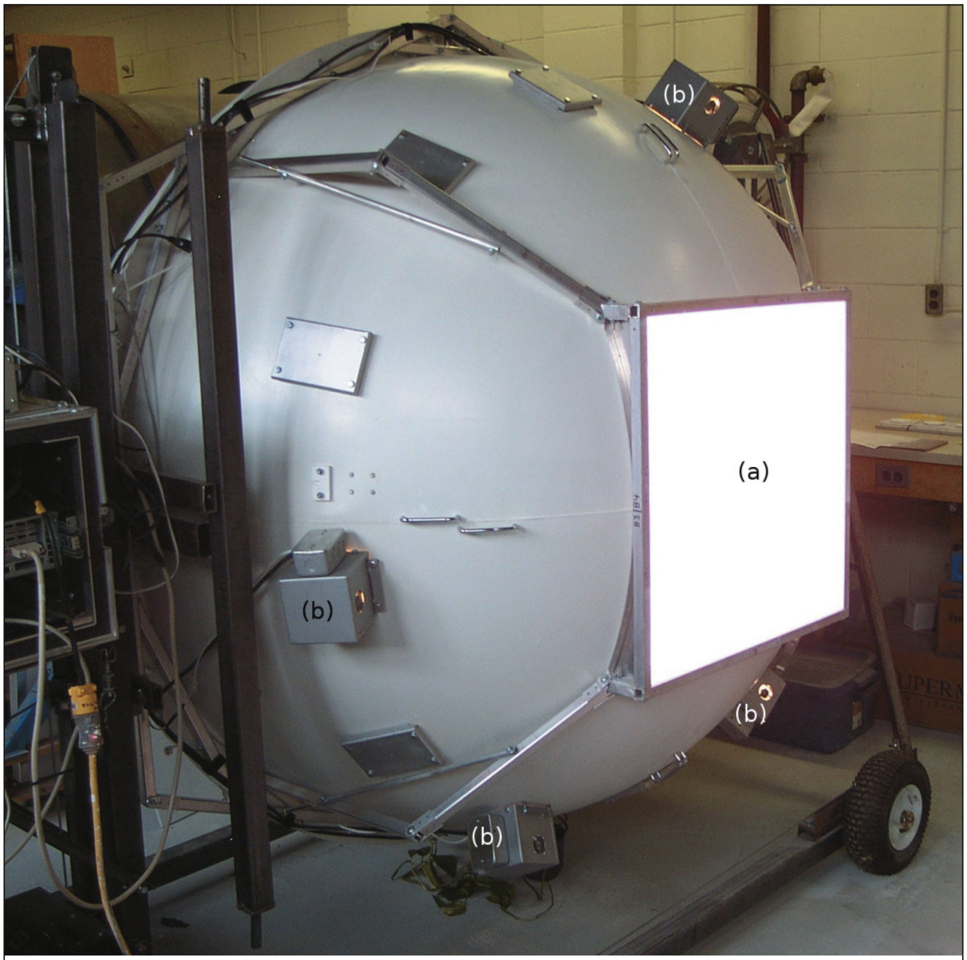

Figure 1. The 1.8 m integrating sphere for which the coating discussed in this paper was developed (Noble, 2006). The integrating sphere is configured as a uniform light source. The sample being illuminated is placed at the sample port (a), and the sphere interior is illuminated by a set of lamps (b). Integrating sphere theory dictates that a spherical interior geometry with a Lambertian coating will disperse incident light such that uniform intensity is experienced at all points of the surface.

reflectance or transmittance of partially diffuse materials. The other significant use of diffusely reflecting surfaces is as calibration targets or standards for sensors.

There are several types of diffuse reflectance materials on the market today, ranging from machinable, solid materials to special coatings applied by the end user and to household, flat white ceiling paint.

Polytetrafluoroethylene (PTFE) is a common diffuse reflector when formed as a microstructured solid. Common examples include Spectralon (Labsphere Inc., North Sutton, N.H.) and Zenith Optowhite (Sphere Optics, Concord, N.H.).[2] These materials typically have very high reflectance, being >98% from 310 to 1700 nm and >95% from 250 to 2500 nm. Generally, a thickness of more than 4 mm is required, and often 10 mm is used. This type of material is also highly durable, washable, and machinable. The disadvantages of these materials are their price, their tendency to absorb oils, and their inability to be applied as a coating.

Barium sulphate ($BaSO_4$) coatings are another common type of diffuse surface. These coatings have the advantage that they can be used on a variety of surfaces, including on shapes that would be difficult to machine out of PTFE. They generally have a narrower effective wavelength range than that of the PTFE materials, being most effective in the 400–1000 nm range, although they still exhibit high reflectance over approximately 300–1800 nm, depending on the exact formulation of the coating. The majority of these coatings are not intended to be applied by the end-user, but rather by the manufacturer. This ensures a high standard of application. Other coatings are sold that are intended to be applied by the end user, providing an in-house application option. This option is also expensive if a large area is to be coated. For example, retailing at US$240, 500 mL of Munsell White Reflectance Coating will cover

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[2] Mention of companies or product brands is included for completeness and does not indicate endorsement by the authors.

approximately 0.13 m^2 (Edmund Optics Inc., 2007). Polyvinyl alcohol (PVAl), which is water soluble, is commonly used as a binder in these coatings.

Flat white paint has also been used as a diffuse reflectance coating in a low-cost uniform light source (Ducharme et al., 1997). This paint was reported to have a reflectance of approximately 85% and was sufficiently diffuse to give the integrating sphere a uniformity of above 98%. This reflectance was more than 10% lower than that of commercially available reflectance coatings, which would translate into an even greater decrease in throughput efficiency for integrating sphere applications. Furthermore, typical white paint is not suitable for applications requiring high reflectance down to 400 nm because of structure of the particular type of titanium dioxide (TiO_2) most commonly used.

A custom coating for an integrating sphere was developed for use in the visible to very near infrared (VIS–VNIR) spectral range (Noble, 2006). There were several reasons for developing an in-house solution. The primary driver was cost, as the area to be coated on a 1.8 m diameter sphere was roughly 10 m^2. The cost to coat this sphere with a commercial, user-applied $BaSO_4$ paint to the recommended thickness was nearly CAN$20 000. The option of shipping the sphere away to be coated was not formally considered, given the costs and inconvenience of shipping and the need to repeat the process in the event of damage to the coating. A second consideration was safety and the lack of suitable facilities for working with relatively large volumes of volatile solvents. User-applied commercial coatings appear to contain a sufficient volume of alcohol in addition to the primary solvent (water) that flammability and ventilation are serious concerns (Labsphere Inc., 1999; Munsell Color Company, Inc., 1999).

When selecting an appropriate binder, safety, availability, cost, and ease of use were the primary considerations. PVAl was excluded due to its water solubility. Binders used early in the development of reflectance coatings included gelatine (Miescher and Rometsch, 1950; Grum and Luckey, 1968) or carboxy-methyl-cellulose (CMC) (Budde, 1960; Grum and Luckey, 1968), both of which are also water soluble and therefore unsuitable for use as a binder in potentially moist or humid environments. Water-borne polyurethane (Varathane Interior Gloss Diamond Water-based Polyurethane, Rustoleum Consumer Brands Canada, Toronto, Ont.) was selected as the binder to use in the coating development. This type of product is readily available at hardware stores, is relatively inexpensive, requires only water for thinning, is nonflammable, has low volatility, and is easy to apply.

The primary pigment used, almost universally, in white paint is TiO_2. The crystal is available in two structures. The rutile form is by far the most common, having better stability and a higher refractive index of 2.73. The less common form is anatase, which has a lower refractive index of 2.55 (DuPont Titanium Technologies, 2002). In a coating, both pigments absorb ultraviolet (UV) light and have a very high reflectance in the visible (VIS) and very near infrared (VNIR) regions of the spectrum. However, the absorption characteristics of the anatase form result in much higher reflectance at blue wavelengths less than approximately 430 nm. As the blue region of the spectrum was of interest, the coating was created using the anatase form of the TiO_2 pigment. In initial testing, $BaSO_4$ was also included as a potential pigment, as it is commonly used in reflectance coatings and does not have the high UV absorption of TiO_2. The $BaSO_4$ tested had poor dispersion characteristics in water, as compared with TiO_2, was found to be more prone to developing surface cracks at high pigment-to-binder ratios, and had lower reflectance above 400 nm for comparable formulations based on the anatase TiO_2. This latter finding was expected, given the difference in refractive index between the two pigments, namely 2.55 for anatase TiO_2 versus 1.64 for $BaSO_4$ (DuPont Titanium Technologies, 2002). Although the $BaSO_4$ coating had higher reflectance below 400 nm, the absorption of UV light by the TiO_2 was considered to have some positive side effects, as it would absorb much of the UV light emitted from the arc lamps used in the system, reducing the potential for eye damage from UV exposure. The anatase TiO_2 was used as the pigment beyond the initial phases of formulation.

The objective of this experiment was to characterize more thoroughly the properties of the coating described by Noble (2006). Specific optical properties of interest were the minimum required thickness for best reflectance, the spectral reflectance, and the bidirectional reflectance factor (BRF) of the coating. The impact of application method on reflectance, the resistance of the coating to wear from cleaning, and the response of the coating to unprotected outdoor exposure were also investigated.

Methods

The coating consisted of anatase TiO_2 pigment (Tronox 110, Tronox Inc., Oklahoma City, Okla.) having a mean particle size of 200 nm, deionized water, and a water-borne polyurethane binder (Varathane Interior Gloss Diamond Water-based Polyurethane). The coating was made by mixing the anatase TiO_2 pigment with distilled water in a ratio of 5 g pigment to 3 mL distilled water at room temperature. The polyurethane binder was then added, 2 mL for every 5 g of pigment used. Additional water was added if required to adjust consistency.

Assessment of minimum required thickness

To assess the minimum required thickness of the coating, ten 27 cm × 27 cm panels of medium-density fibreboard were prepared with a layer of flat black spray paint. The panels were numbered one through ten, and a number of coats corresponding to the panel number were applied using a paint roller. The minimum dry time between coats was 3 h. Thickness measurements of the coating layer on each panel were taken several days after the coating application was complete for all panels. The coating was removed at five spots on each panel, and the difference in thickness between the uncoated patch and adjacent coated area was measured. This was originally to be

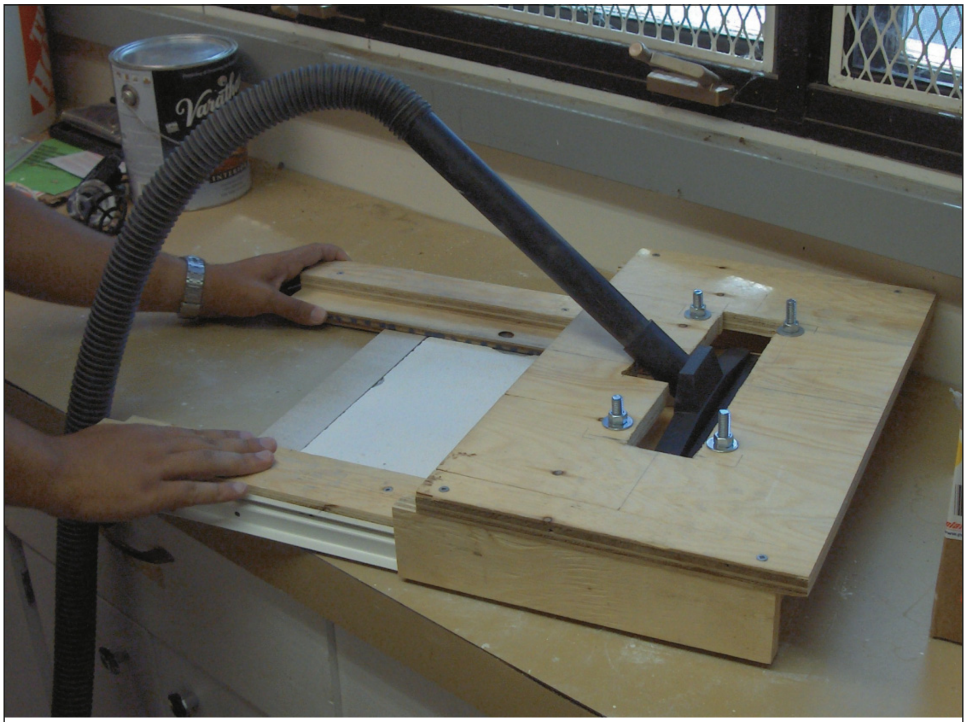

Figure 2. Abrasion apparatus.

done based on panel thickness at the five locations prior to coating, but the measurement apparatus used for the precoating measurements gave inconsistent results due to a mounting error. The thickness measurements were repeated at the same five locations on each panel, and the reflectance was measured at each point using a Fieldspec FR spectroradiometer (Analytical Spectral Devices, Boulder, Colo.) and LI-COR 1800 integrating sphere (LI-COR, Lincoln Nebr.). The effective available wavelength range was between 350 and 1800 nm. Plots of average reflectance versus average thickness at several wavelengths were used to determine the required thickness for the maximum reflectance of the coating over the relevant spectrum.

BRF measurement

The BRF was measured using the University of Lethbridge Goniospectrometer (ULGS), equipped with the ASD Fieldspec spectroradiometer using a 5° foreoptic (Coburn and Peddle, 2006). The sample was illuminated at 1.5° from normal with a 50 W quartz-halogen lamp with an aluminized MR-16 style lamp (model L521, Gilway Technical Lamp, Peabody, Mass.) at a distance of 0.6 m from the sample. Measurements were taken at 10° intervals from –60° to +60° along the zenith arch. Thus, 13 measurements, each the average of 25 readings returned by the spectroradiometer, were taken for each panel.

A calibrated Zenith Optowhite diffuse reflectance target was assumed to exhibit Lambertian reflectance for the purposes of comparing the angular distribution of the light reflected from the samples. To isolate the reflectance (measured with the integrating sphere) from the angular distribution, each set of measurements was normalized by the sum of values for the set. The value at each angle was then divided by the value at the corresponding angle for the sum-normalized Zenith reference. When plotted against angle, the curve of the Zenith material would be a horizontal line with a value of 1.0.

Effect of application method

The impact of application method on reflectance, angular distribution of light, and how these may change with wear was investigated for three common paint application methods, namely brushing, rolling, and spraying. Five panels for each application method were coated to at least the required minimum thickness. The reflectance of each coated panel was measured using the spectroradiometer and integrating sphere, and the angular distribution of reflected light was tested using the ULGS.

These coatings were then tested for their wear characteristics with respect to reflectance and light dispersion by abrading the panels and repeating the measurements. A number of standard scrub-abrasion tests have been developed (Kirsch et al., 2001). These tests involve a recurrent back-and-forth motion with an abrasive material or brush to evaluate the resistance of a coating to scrubbing. The standard tests measure, in one way or another, how much scrubbing a coating can withstand before it wears through. This coating was not developed to have good scrub resistance as compared to that of a typical paint or coating, and because of its very high pigment to binder ratio it would not stand up well to these tests, nor would these tests provide the desired information, namely the degree to which lightly brushing the coating to remove dirt or dust would affect

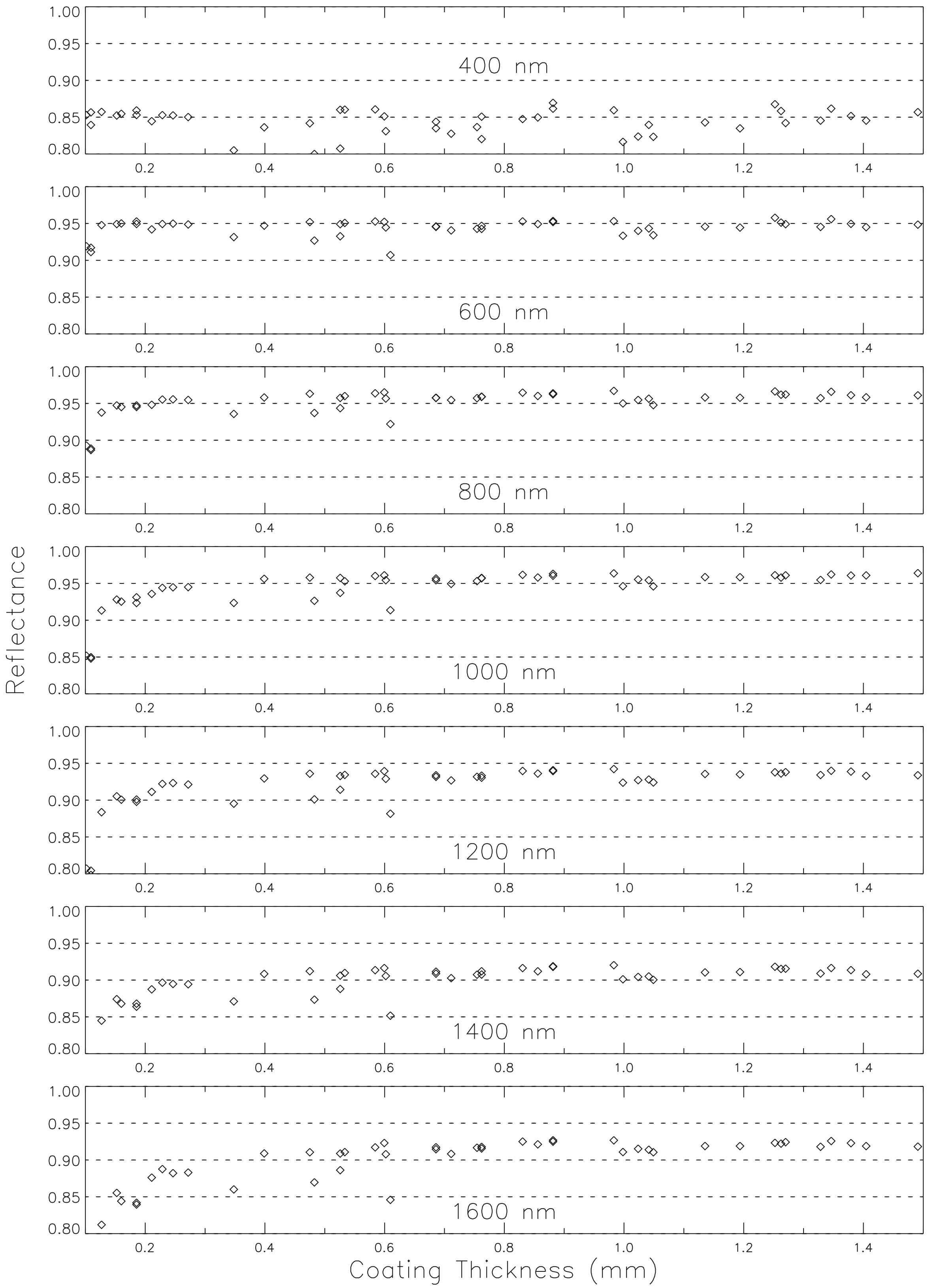

Figure 3. Plots of reflectance versus coating thickness for selected wavelengths between 400 and 1600 nm.

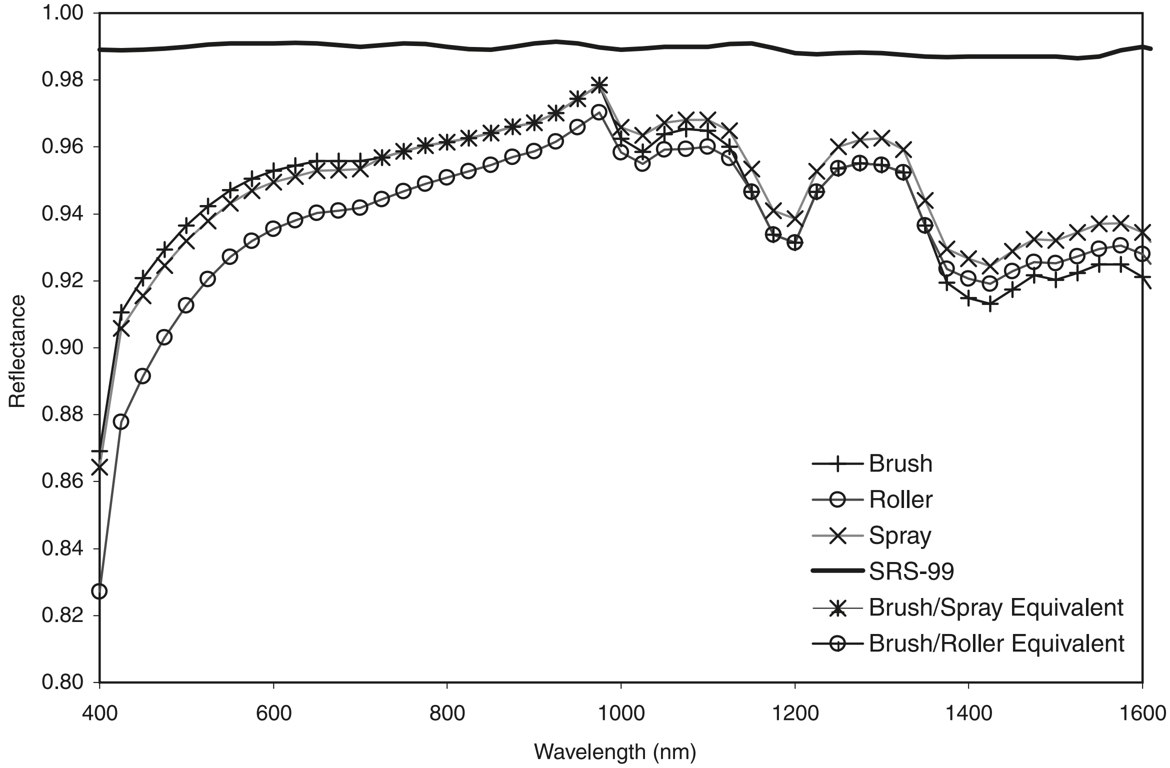

Figure 4. Comparison of mean reflectance levels between application methods. Non-overlapping points are significantly different at the 95% confidence level.

the BRF of the coating. To this end, a test loosely based on the parameters described by Kirsch et al. (2001) was developed.

The testing device consisted of a base, into which the panels were placed, and a frame mounted on rails attached to that base (**Figure 2**). A floor brush attachment for a wet–dry vacuum (part 90604, Shop-Vac Corporation, Williamsport, Pa.) was mounted on the frame. The base was placed on top of the panel under test, and the frame and attached brush were moved back and forth at a fixed height across the panel. The bristles on the brush provided the abrasion, and the connected vacuum prevented excessive dust build-up caused by the accelerated test. Three panels for each of the application methods were placed under the testing device for 5, 25, and 50 cycles, with a single cycle consisting of two brush passes: one in the forward direction, and one returning to the starting position. The BRF of each test panel was measured after each round of abrasion.

Weathering effects

Accelerated weathering tests are unquestionably the most controversial area of the study of paints and coatings (Ellinger, 1979). There are dozens of variations on how to run an accelerated weathering test, and there is very little agreement on which method is best (Blakey, 1985). With that in mind, and the lack of equipment required for an accelerated test, it was decided that a real-time test was the best route to follow.

Three panels were prepared and placed outdoors in an unshaded area between 4 August and 27 September 2006. Panels were mounted on a south-facing shed roof at an angle of 60° from the ground. Based on data from the Lethbridge Airport weather station (Meteorological Service of Canada 2006: Lethbridge AWOS A, Climate ID 3033884, 49°37′N, 112°48′W, elevation 928.70 m), the average temperature for this period was 15 °C, with a total precipitation of 30.5 mm. This period included overnight freezing temperatures and frost. Panel reflectance was measured after 2 weeks and at the end of the exposure test (approximately 1.5 months). Three reflectance measurements were made on each panel and time period using the spectrometer and integrating sphere.

Results and discussion

Thickness

The relationships between coating thickness, wavelength, and reflectance can be observed in the plots of **Figure 3**. The point at which the slope of the curve for each wavelength becomes approximately zero was considered to be the minimum required thickness for that wavelength, with the greatest minimum thickness of all wavelengths being the overall minimum application thickness. As expected, a correlation was found between coating thickness and reflectance, with reflectance increasing with increasing thickness. This was much more pronounced at longer wavelengths, and less evident at shorter wavelengths. It is likely that the thinnest coating was thick enough to achieve infinite

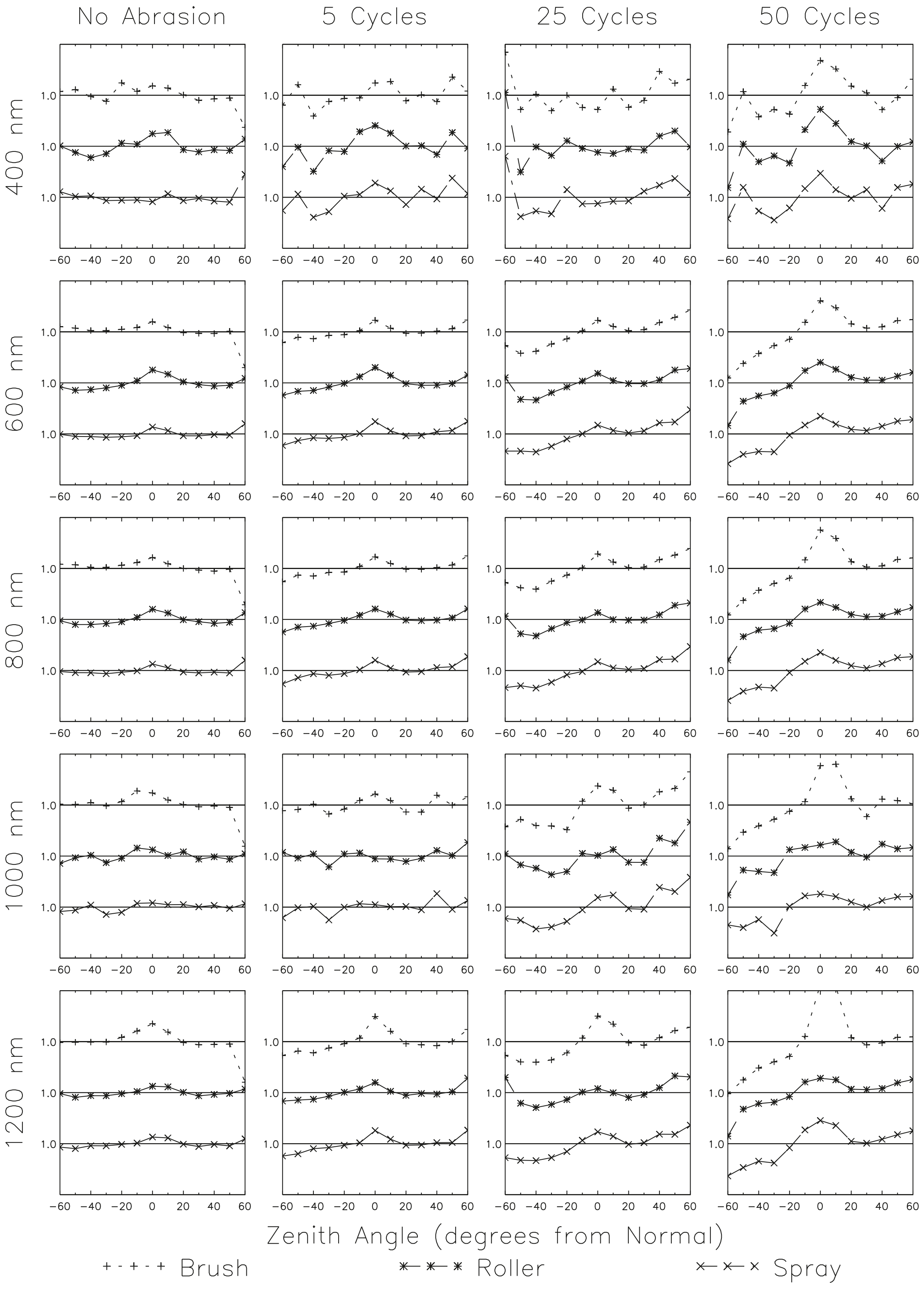

Figure 5. BRF with respect to wavelength and abrasion (0, 5, 25, and 50 cycles). Curves were normalized by area and referenced to Zenith reflectance material (straight solid lines), which was assumed to be ideally Lambertian for purposes of comparison. Plots are offset from each other by 0.25. Plots compare relative reflectivity at a particular zenith angle (plotted on *x* axis) and are independent of reflectance.

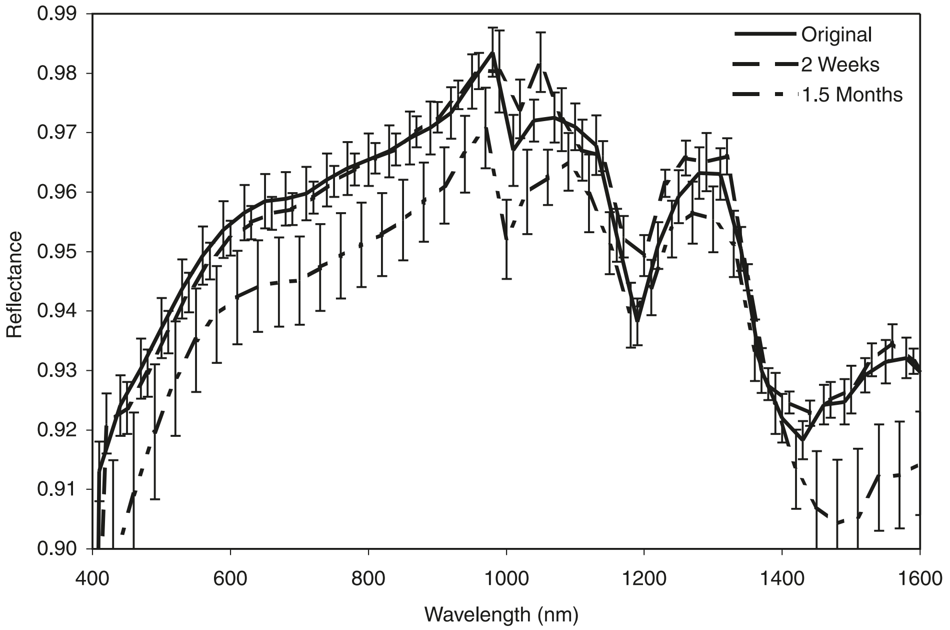

Figure 6. Reflectance measurements from panels after no weathering, 2 weeks of weathering, and 1.5 months of weathering. Error bars indicate ±1 standard deviation, based on three repeated measurements on three replicate panels.

reflectance at the lowest wavelengths (400–550 nm), and a thicker coating was needed to achieve the infinite reflectance at higher wavelengths, as the light penetrated more deeply. A minimum thickness of 0.7 mm was judged to give an infinite reflectance over the measured spectrum of 400–1750 nm. Coatings thicker than this minimum thickness did not demonstrate a loss of reflectance.

Application methods

A plot comparing the effects of application method on reflectance is shown in **Figure 4**, with all three application methods compared to a calibrated white Spectralon standard, labelled SRS-99. Non-overlapping points are significantly different at the 95% confidence level, based on pairwise *T* tests. Between 400 and 1000 nm, brush and spray applications resulted in greater reflectance than roller application by less than 4.5%. No significant difference in reflectance was found between brush and spray application in the 725–1000 nm range. Beyond 1000 nm, spray application resulted in the highest reflectance, but by less than 1% reflectance. On the basis of reflectance alone, either brushing or spraying would be the preferred application method for use between 400 and 1000 nm. At longer wavelengths, spraying would be preferred to brushing or rolling.

BRF and abrasion

The data collected for the BRF were compared against the measured BRF of a Zenith Optowhite diffuse reflectance target, which was assumed to be a perfectly diffuse reflector. These results are shown in **Figure 5** for no abrasion and for 5, 25, and 50 abrasion cycles and at five illustrative wavelengths. A straight, horizontal line with a value 1.0 would indicate a dispersion pattern identical to that of the Zenith Optowhite material. Points above or below the line indicate more or less light, respectively, reflected at a particular angle than would be expected from a diffuse reflector. In general, the reflectivity became more specular with increased abrasion levels. At zero abrasion, the application by spray was comparatively more diffuse than the other methods. The directionality of the brushed coatings increased slightly with increasing wavelength, whereas the directionality of roller-applied coating decreased with the same increases in wavelength. The impact of abrasion was most pronounced at longer wavelengths, with directionality generally increasing for all application methods with increased abrasion. Brush-applied coatings were more affected by abrasion than were the spray- and roller-applied coatings. The panels painted with the brush also had less consistent results. The differences between the roller-applied and sprayed panels were small, and the better choice depended on wavelength. At wavelengths shorter than 1000 nm the sprayed panels displayed slightly more diffuse characteristics. The results from the roller-applied panels were slightly more consistent above 1000 nm and slightly more diffuse than the sprayed panels at longer wavelengths.

Weathering

Average panel reflectances before weathering after 2 weeks and after 1.5 months are shown in **Figure 6**. Little difference in reflectance was evident after 2 weeks; the variability actually appeared to decrease slightly. After 1.5 months of exposure, a reduction of approximately 2% reflectance was observed, and measurements were more variable than those taken from unweathered panels. Physically, the coating was more brittle and prone to chipping, chalking, and falling off the panel after weathering, but not to a degree that made the panels significantly more difficult to handle.

Conclusions

The coating was found to require a thickness of greater than 0.7 mm for maximum reflectance across its effective wavelength range. Overall, spray application, followed by roller application, were judged to be the preferred methods of application for integrating spheres on the basis of having more Lambertian bidirectional reflectance factor (BRF) characteristics as compared to brush application. The BRF of coatings that were sprayed or rolled on was also less affected by abrasion as compared with that of the brushed-on coating. To date, the coating has performed well in the large uniform light source for which it was developed, with uniformity values approaching 98% between 400 and 850 nm (Noble, 2006).

The suitability of the coating for outdoor, remote-sensing work will depend on the environment and duration of exposure. It may be suitable for use in fieldwork as a transfer standard, allowing the considerably more expensive primary standard to remain in a safe location. The coating is inexpensive enough that large, highly diffuse reference targets could be constructed at a relatively low cost. With a maximum decrease in reflectance of less that 2% in one and one-half months, test results indicated that such a target could be expected to remain stable under continuous exposure for a period of several weeks in a dry environment. The coating was observed to absorb water if it was present. Water in the coating would be expected to lower reflectance in water-absorption bands, however this was not tested. This may be of concern at longer wavelengths, particularly in the water-absorption bands, and may introduce variability if measurements under different moisture conditions are to be compared.

Acknowledgements

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