**Optical properties of a long dynamic range chemical UV dosimeter based on solvent cast polyvinyl chloride (PVC)**

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# Abstract

The dosimetric properties of the recently introduced UV dosimeter based on 16 µm PVC film have been fully characterized. Drying the thin film in air at 50 ºC for at least 28 days was found to be necessary to minimise the temperature effects on the dosimeter response. This research has found that the dosimeter response, previously reported to be mainly to UVB, has no significant dependence on either exposure temperature or dose rate. The dosimeter has negligible dark reaction and responds to the UV radiation with high reproducibility. The dosimeter angular response was found to have a similar pattern as the cosine function but deviates considerably at angles larger than 70º. Dose response curves exhibit monotonically increasing shape and the dosimeter can measure more than 900 SED. This is about three weeks of continuous exposure during summer at subtropical sites. Exposures measured by the PVC dosimeter for some anatomical sites exposed to solar radiation for twelve consecutive days were comparable with those concurrently measured by a series of PPO dosimeters and were in line with earlier results reported in similar studies.

# Introduction

Chemically-based ultraviolet (UV) radiation dosimeters have been widely used in personal UV exposure measurements since 1976. Despite the development of the electronic ultraviolet (EUV) dosimeters, chemical UV dosimeters maintain their suitability in human exposure research as simple and economical UV monitors that require no power and can be miniaturised [1, 2]. The most commonly used chemical UV dosimeters are the polysulphone (PS) [3] and polyphenylene oxide (PPO) [4, 5] dosimeters. The PS dosimeter has long been employed in quantifying human exposure to solar UV in different environments during various activities [6-15]; and effectively used in evaluating UV minimizing strategies [16-18]. The PPO dosimeter, employed later than PS, has also been successfully utilised in measuring UV exposures in terrestrial and aquatic environments [19-21]. The dynamic range of PS and PPO is limited to up to eight hours and five days respectively of exposure to solar UV radiation. Consequently, the dosimeters have to be replaced periodically for longer measurements, increasing the cost, effort and margin of error of the measurements.

Quantifying long-term personal UV exposure has a great importance, particularly for studies of latent outcomes of exposure to UV radiation [22]. It is well known that cumulative exposure throughout lifetime is a risk factor for a number of cutaneous diseases, including photaging [23] and some types of skin cancer [24], and ocular diseases such as pterygium and cataracts [25, 26]. A long-term personal UV dosimeter takes into account the day-to-day variations in exposure related to personal activities and environmental conditions. Therefore, prolonged measurements will be more accurate and reliable than the results obtained using short dynamic range dosimeters.

A new dosimeter based on a thin film of polyvinyl chloride (PVC) cast from tetrahydrofuran (THF) solution has recently been introduced with the ability of measuring at least two weeks of continuous exposure to solar UV radiation at subtropical sites [27]. Further research has identified the best PVC/THF mixing ratio to prepare the dosimeter as 10% and the ideal thickness of the dosimeter as 16 µm [28]. In the same research, the dosimeter responsivity was determined to be mainly to the UVB waveband and thereby the dosimeter can be calibrated for erythemally weighted exposures. Dose response curves were established by relating the UV-induced absorbance change at the 1064 cm-1 peak with the incident exposure. However, to achieve accurate dose measurements, the basic dosimetric properties reported in the literature [29, 30] have to be studied. This paper reports on the investigation of the key aspects of the PVC dose response: reproducibility, temperature independency, dose rate independency, angular dependence, backscattering effect, threshold dose and dark reaction. Seasonal dose response curves have also been provided and a field trial has been conducted to test the dosimeter validity.

# Materials and Methods

## Drying time determination

Pure polyvinyl chloride (PVC) does not absorb any radiation above 220 nm [31] and thereby no changes should occur in PVC during its exposure to UV radiation. However, PVC is known to be degraded on exposure to UV radiation mainly because of impurities, arising from either thermal treatment during processing or added ingredients, which catalyse photodegradation processes or absorb UV radiation to form radicals that initiate further reactions [32]. PVC thin films prepared by casting from tetrahydrofuran solution for use in UV dosimetry have not been treated thermally and contain no additives. Therefore, the UV-induced changes are assumed to be a result of the influence of residual tetrahydrofuran, which is considered as a photosensitiser [32] and responsible for the 1064 cm-1 peak in the infra red (IR) absorption spectra of solvent cast PVC samples [33]. Solvent residues remain even when the films are dried at high temperatures (up to 120 ºC) for extremely long times [34]. Maláč et al. [34] dried solvent cast PVC samples at 50 ºC in air and found that the amount of residual tetrahydofuran in the samples decreases with time up to about 25 days and then remains nearly constant. The response of PVC dosimeters dried for various times to UV radiation, therefore, has to be investigated to determine if it is necessary to dry the PVC thin films and for how long.

Ninety solvent cast PVC dosimeters of thickness of about 16 µm were prepared as described elsewhere [28]. The absolute absorbance of the dosimeters at 1064 cm-1 (hereafter only “absorbance”) was measured immediately using a Fourier Transform Infrared (FTIR) spectrophotometer (IRPrestige-21/FTIR-8400S, Shimadzu Co., Kyoto) and found to be 0.249 ± 0.012. The dosimeters were then dried at 50 °C in air for about four weeks and their absorbance was measured periodically. During the drying process, eight dosimeters were removed each five days and exposed to a broadband UV radiant exposure of 200 kJ/m2 supplied by a UV fluorescent lamp (model Philips TL40/12, supplier Lawrence and Hansen, Toowoomba) that emits radiation primarily in the UVB region of the UV spectrum . Four of the dosimeters were exposed at 25 °C and four at 45 °C. The spectral irradiance at the dosimeters’ site  was measured with a calibrated scanning spectroradiometer (model DMc150, Bentham Instruments Ltd, Reading UK) and the radiant exposure  received by the dosimeters was calculated by:

 (1)

where  is the wavelength increment and  is the exposure time. The corresponding absorbance change  was calculated by

 (2)

where  is the pre exposure absorbance and  is the post exposure absorbance. The average deviation of the lamp output during the exposure and the irradiance uniformity at the dosimeters’ site were about 3% and 3% respectively.

## Reproducibility

The reproducibility is one of the quantities that define the dosimeter reliability. Equal radiant exposures from the same source should induce the same measured response. Any errors related to the film reproducibility will affect the results and have to be quantified and taken into account during the measurement process. To quantify the reproducibility of the measurements, ten dosimeters were irradiated evenly by the fluorescent UV lamp for 20 days, receiving a total radiant exposure of 27 MJ/m2 of broadband UV radiation. The mean absorbance change of the dosimeters was determined at various intervals, and the average deviation from the mean was obtained and plotted as a function of the radiant exposure.

## Temperature independency

Chemical dosimeters employed in solar UV radiation measurements experience different ambient temperatures. Therefore, the thermal stability of the dosimeter has to be ensured and the temperature range of the dosimeter validity should be identified. To investigate the effect of temperature on the response of the PVC dosimeter to UV radiation, five groups of 16 µm PVC dosimeters, previously dried at 50 °C for four weeks, were sequentially exposed at 5 ºC, 12 ºC, 20 ºC, 30 ºC, 40 ºC, and 45 ºC to radiant exposures of 200 kJ/m2 using the fluorescent lamp. Irradiation at different temperatures was achieved by controlling the air flow temperature, except for 5 ºC in which a water bath containing a mixture of ice and water was used. The temperature error was estimated to be ±2 ºC. The IR absorbance was measured before and after the exposure to allow comparison of the absorbance change of the dosimeters exposed at different temperatures.

## Dose-rate independency

Chemical UV dosimeters measure the integrated radiant exposure (dose). Therefore, the UV-induced response within the dosimeter should be independent of the rate of the received dose (irradiance), i.e., equal doses are supposed to induce the same response regardless of the dose rate and duration of exposure. The dose rate dependence of the PVC dosimeter was studied by exposing seven groups of PVC dosimeters sequentially to the fluorescent lamp at different distances from the lamp for different durations of exposure, so that all groups received an equal broadband UV radiant exposure of 2 MJ/m2. The distance between the dosimeters and the lamp ranged between 6 cm and 34 cm. The UV irradiance as measured by the Bentham spectroradiometer at the exposure site was between 12.9 W/m2 and 2.9 W/m2; and the required exposure time ranged between 43 and 193 hours. The average absorbance change of each group was measured, normalised and plotted against the corresponding irradiance. For each exposure, an additional three dosimeters covered with cardboard were employed as control dosimeters. The covered dosimeters were placed at the exposure site to ensure that they were at the same temperature as the exposed dosimeters.

## Angular dependence

The angular dependence of the dosimeter is the variation in its response with the angle of incidence of incoming radiation. The maximum irradiance of a stable beam of radiation corresponds to angle of incidence, and reduced by a factor of  with increasing angle [35]. Therefore, for an ideal UV dosimeter, the response to the change in the angle of incidence of the beam should be a cosine function. To quantify any differences of the angular response of the PVC dosimeter from the cosine function, nine sets of PVC dosimeters were irradiated by a collimated UV beam at nine different angles of incidence. The irradiation source was a UV solar simulator (19160-1000, Newport Co., California, USA) combined with an exposure controller (model 68945, Newport Co., California, USA) to minimise variations in the lamp output. The simulator provides a stable collimated beam of 5 cm×5 cm with measured irradiance uniformity across the entire exposure area of about 5%. Batches of four PVC dosimeters were irradiated sequentially at angles ranging from 0º to 80º at intervals of 10º. The exposure lasted for 24 hours at each angle. The average absorbance change of the dosimeters corresponding to each angle was measured and then normalised for the comparison with the cosine function. The deviation of the dosimeter angular response from the ideal cosine response was calculated for each angle.

## Backscattering effect

Butson et. al. [36]have reported a variation of up to 19% in the measurements of UV exposure obtained with a UV dosimeter based on a radiochromic film due to various background materials. This discrepancy is assumed to be associated with backscattered radiation reflected off backing material. Therefore, the effect of the background material and colour on the response of PVC dosimeters to UV radiation was examined. White, black and purple papers along with black polyethylene and glossy wood were employed as backgrounds for five batches of PVC dosimeters. The dosimeters were exposed evenly to the fluorescent UV lamp for 100 hours, providing 5 MJ/m2 of broadband UV radiant exposure at the dosimeters’ site. The UV-induced absorbance change of the dosimeters was measured and normalised for the comparison.

## Erythemal dose response curves

The erythemal dose response curves of the PVC dosimeter were determined at the University of Southern Queensland, Toowoomba, Australia (latitude 27.6 ºS) in Autumn (23 April - 29 May 2012), Winter (23 July - 28 August 2012), Spring (10 September - 8 October) and Summer (5 February - 22 February 2013). For each season, five dosimeters have been exposed on a horizontal unshaded plane near an erythemally weighted UV meter (model 501 Biometer, Solar Light Co. PA. USA), that records the cumulative erythemal exposure each five minutes. The Biometer was calibrated to the Bentham spectroradiometer each season. The angular response of the Biometer is assured by the manufacturer to be within 5% from ideal cosine for all incident angles. The dosimeters were removed only for the absorbance measurements (15 minutes) and were not brought indoors at night. The absorbance change of the dosimeters was measured at different intervals and related to the corresponding exposure dose to construct dose response curves. During the summer calibration, three covered dosimeters (control dosimeters) were placed at the exposure site to investigate any effects of the ambient temperature on the UV-induced changes within the dosimeter.

## Threshold dose

The threshold dose is the smallest dose that can induce a measureable response within the dosimeter. The erythemal dose response curves obtained for the PVC dosimeter indicate that the dose capacity of the dosimeter extends to more than 900 SED. However, the response to small doses does not exactly match the general trend of the data. Further investigation of the dosimeter response to small doses has been carried out using the solar simulator. A set of four PVC dosimeters was evenly exposed to a total radiant exposure of 6 SED, during which the absorbance change was measured regularly.

## Dark reaction

Previous research has reported changes in some chemical UV dosimeters’ response after the UV exposure has been terminated [3, 5, 37, 38]. This behaviour is commonly known as the dark reaction. The presented PVC dosimeter aims to measure weeks of exposure to solar UV radiation and thereby will experience nocturnal periods during the measurements. In addition, the readout process is not always available immediately after exposure and the dosimeters may have to be stored for a while. The post exposure behaviour of the PVC dosimeter was investigated by exposing 16 PVC dosimeters to a radiant exposure of 3.5 MJ/m2 supplied by the fluorescent lamp. After measuring the UV-induced absorbance change, the dosimeters were divided into four groups and maintained in a UV free environment at -15 ºC, 0 ºC, 15-20 ºC and 40 ºC respectively. The absorbance change was measured at different times during the storage and plotted against time for each temperature.

##  2.10 Field trial

An outdoor test was carried out to evaluate the use of the PVC dosimeter in measuring personal exposure in a field trial to solar UV during long periods. The results were compared with those concurrently measured by a series of PPO dosimeters. PVC and PPO dosimeters were employed for monitoring the exposure received by five anatomical sites using four upper-body manikins rotating at a speed of one revolution per minute. Dosimeters of both types were attached to the following sites: vertex, nose, shoulder, chin and neck. The exposures were carried out for 12 consecutive days during summer 2013 at a private property near the University of Southern Queensland. The site of the exposures was an unshaded lawn surrounded by a house and fence, with partial shading before 8:00 and after 18:00 EST. The dose response curves were determined at the same time by exposing a number of PVC and PPO dosimeters on a horizontal unshaded plane near the erythemally weighted UV meter and recording the UV-induced response as a function of the UV exposure. The UV-induced response of the PPO dosimeter was quantified by the change in the absorbance at 320 nm [5] measured using a spectrophotometer (model UV-1601, Shimadzu Co., Kyoto, Japan). Due to the fact that PPO dosimeters are saturated after about five days of exposure to solar UV radiation [5], the measuring and calibrating PPO dosimeters were replaced after each four days of exposure. At the end of the exposures, the anatomical exposure measured by one set of PVC dosimeters (10 dosimeters) was compared with that measured using three sets of PPO dosimeters (30 dosimeters).

# Results

## Drying time determination

Figure 1 shows the absorbance of the PVC dosimeters dried at 50 °C as a function of the drying time. The error bars represent the standard error of the measured absorbance. There was a gradual absorbance reduction with a decreasing rate of change. Although the absorbance curve has not eventually levelled as expected [34], the rate of absorbance decrease reached its minimum after about 3 weeks of drying. The absorbance dropped to 50% of its initial value after four weeks of drying. The difference between the absorbance change of PVC dosimeters exposed to 200 kJ/m2 at 25 ºC and 45 ºC decreased with the increase of drying time (Figure 2). The error bars represent the standard error of the normalised absorbance change as calculated by the error propagation formulae. The absorbance change of the undried dosimeters exposed at 45 ºC was about 30% higher than those exposed at 25 ºC. This difference dropped to just about 5% for those dosimeters dried for 25 days, indicating that drying the dosimeters for about 25 days at 50 ºC will minimise the temperature dependence of the dosimeters.

## Reproducibility

The average deviation from the mean absorbance change of the ten dosimeters was 3-5% for broadband UV exposures up to 2.5 MJ/m2 and dropped to ≤ 2% for higher exposures (Figure 3). The y error bars represent the standard error of the measured absorbance change, they are shorter than the dimensions of the associated symbol and do not appear clearly on the graph, while the x bars represent 3% variation in the output of the lamp..

## Temperature independency

The dosimeters exposed at various temperatures showed no significant difference in their behaviour and the response seems to be independent of temperature. Figure 4 shows the normalised response of PVC dosimeters exposed over the range 5-45 ºC. The average deviation from the mean response was about 4.5 %.

## Dose-rate independency

The response of PVC dosimeters exposed to an equal radiant exposure accumulated from different irradiances with different durations of exposure is given in Figure 5. The average deviation from the mean response was about 6 %.

## Angular dependence

The response of the PVC dosimeter as a function of the angle of incidence of UV radiation is shown in Figure 6 together with the cosine function and the corresponding cosine error. The y error bars represent the standard error of the normalised response as calculated by error propagation formulae while the x error bars represent an estimated constant angular alignment error of 1º. The measured cosine error was less than 6.5% for angles up to 40º, increasing to 16% at 50º and reaching its maximum of about 40% at higher angles.

## Backscattering effect

The response of PVC dosimeters evenly irradiated using different backgrounds is shown in Figure 7. In general, darker backgrounds had less effect on the absorbance. This is in agreement with previous research [39, 40] that reported less UV reflectivity of dark colours than the light ones. The maximum response was for the white paper background. Responses for white and purple backgrounds differed by about 30%. In addition, a slight effect of the material type is noticed by the 5% discrepancy in the response of dosimeters irradiated with black polyethylene and black paper. The results indicate that similar materials and colours should be employed for both calibrating dosimeters and measuring dosimeters to avoid any backscattering effect.

## Erythemal dose response curves

Figure 8 shows the erythemal dose response curves of the PVC dosimeter obtained for different seasons. The best fit of the data for all curves is almost similar and very close to linear. Control dosimeters used during summer calibration (when the temperature is the highest) showed an absorbance change of ±1%, except one point when the change was 2%. This change is within the experimental error of the absorbance measurements, indicating that there was no significant effect of ambient temperature on the measured response. The maximum measured cumulative exposure was about 900 SED and obtained during the spring calibration (28 days of exposure) with no signs of saturation or substantial deviation from the general trend of the curve, indicating that the dosimeter is able to measure even higher doses. The combined data of all seasons is shown in figure 9. The curve could be used as a standard dose response curve for the different seasons.

## Threshold dose

A fluctuation in the response is noticed for doses less than 2 SED (Figure 10). After that, the response started to change monotonically. A dose of 3 SED can be considered as the threshold dose of the dosimeter.

## Dark reaction

Figure 11 illustrates the percentage change in ∆A% of four PVC batches maintained after exposure at different temperature. The change was within the experimental error ± 2% and independent of temperature.

##  3.10 Field trial

The doses received by the five anatomical sites during the study period as measured using the PVC and PPO dosimeters are shown in Figure 12. The agreement between the results was best for the vertex, nose and shoulder. Although there was a slight discrepancy between the two dosimeters for doses measured at the chin and back of the neck, measurements obtained by the PVC dosimeter agree with the generalization of earlier studies where the shoulders generally receive two-thirds of the dose relative to the vertex while vertical sites of the body receive roughly half of the vertex dose [29, 41-43]

# Discussion and conclusion

The results presented in this research demonstrate that the PVC film dosimeter fulfils, in general, the criteria required for a reliable UV dosimeter. The 16 µm PVC films prepared from 10% PVC/THF solution and dried in air at 50 ºC for 28 days showed a high reproducibility in their response to UV exposure. The response was within 2.5% of its mean, with a maximum deviation of 5% for exposures less than 2.5 MJ/m2 (Figure 3). In addition, there was no significant dependence of the dose response on exposure temperature over the range 5-45 ºC (Figure 4). The average deviation from the mean response was 4.5 %, which is an acceptable value considering the 3% variation in the lamp output, 5% reproducibility and an uncertainty of about 1.5% for the absorbance measurements. Likewise, no clear relationship was found between the dose response and dose rate for the tested irradiances (3 W/m2 - 13 W/m2) (Figure 5). The response over this range of irradiances deviated within an average of 6% from the mean. Additionally, the general shape of the angular response of the PVC film was similar to the cosine function (Figure 6). The difference between the curves was less than 6.5% for angles up to 40º, but it reached 40% for angles ≥70º. Furthermore, the dark reaction induced response was negligible and independent of temperature (Figure 11). The study of the dosimeter’s sensitivity to small doses showed that an erythemal dose of 3 SED seems to be the threshold dose of the dosimeter (Figure 10). However, high threshold dose is not necessarily a disadvantage, as the dosimeter is proposed for long term exposures. Additionally, the results demonstrated the necessity of using the same background material for both calibrating and measuring dosimeters to eliminate any response due to the backscattering radiation (Figure 7). Finally, the dosimetric characteristics of the PVC dosimeter are comparable with those of PPO and PS dosimeters (Table 1) with a far higher dose capacity for the PVC dosimeter.

The establishment of the PVC dose response curves underlined not only the high dose capacity of the dosimeter, but also the suitability of the dosimeter to be used in a range of environmental conditions. The dose response curves indicate that the PVC dosimeter can measure more than 900 SED of exposure. This is equivalent to about three weeks of continuous exposure during summer at subtropical sites.

The anatomical exposure measurements obtained by the PVC dosimeter over 12 consecutive days agreed well with the results reported in earlier similar studies, supporting the positive overall evaluation of the PVC dosimeter.

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Figure 1: Change of absolute absorbance at 1064 cm-1 of PVC dosimeters as a function of drying time at 50 ° C.

Figure 2: Normalised absorbance change of PVC dosimeters exposed to 200 kJ/m2 at 25 °C and 45 °C after being dried for different periods at 50 ºC.

Figure 3: The average deviation from the mean of ten dosimeters’ absorbance change as a function of the radiant exposure.

Figure 4: The response of PVC dosimeters as a function of the temperature during exposure.

Figure 5: The response of PVC dosimeters exposed to 2 MJ/m2 of broadband UV exposure as a function of the supplied irradiance.



Figure 6: The response of PVC dosimeters as a function of the angle of the incident beam (dashed line) compared with the ideal cosine function (thin line). The bar chart shows the deviation of the PVC angular response from the cosine function.

Figure 7: The response of PVC dosimeters irradiated evenly over different backgrounds.



 Figure 8: Solar erythemal dose response curves of the PVC dosimetr. The summer curve includes the response of the control dosimeters.

Figure 9: Combined dose response curve for the four seasons.

Figure 10: The response of PVC dosimeter to small doses.

Figure 11: The dark reaction induced change of PVC dosimeters kept at different temperatures as a function of time.

Figure 12: Doses received by five anatomical sites due to exposure to solar radiation for 12 days as measured by PVC and PPO dosimeters.

|  |  |  |  |
| --- | --- | --- | --- |
|  | PVC | PPO [5] | PS [3, 44-47] |
| Sensitivity | Primarily to UVB  | Primarily to UVB | Primarily to UVB |
| Dose Capacity (SED) | > 900  | ~ 300  | ~ 30 |
| Reproducibility(response variation)  | 5 % | 6.5 % | 5 % |
| Temperature dependency | Independent4.6 % variation in the response within the range 5-45 ºC | Independent< 2% variation in the response within the range1.5-50 ºC | Independent |
| Dose rate dependency | Independent6.4 % variationwithin the range 3-13W/m2 | Independent 4 % variation in the response within the range  2.1-11.7 W/m2+ Slight dependency 13 % variation in the response within the range 1-3.7 W/m2 | Independent5 % variation in the response within the range 0.56-3.4 W/m2 |
| Angular response (% deviation from the cosine function) | < 6.5 % for angles up to 40ºincreasing to up to 40% for angles ≥ 60º | < 6.2 % for angles up to 40º increasing to up to 13.2 % for angles ≥ 60º | Independent for angles up to 70º |
| Dark reaction induced change after exposure  | Negligible No clear trend | ~ 3.4 % per nightDependent on temperature | 4 % after 24 hours5 % after a week |

Table 1: A comparison of the dosimetric properties of the PVC, PPO and PS dosimeters.