



Personal monitoring of cutaneous vitamin D₃ production through a printable UV molecular dosimeter

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ABSTRACT

This paper presents a promising solution to monitor the cutaneous production of vitamin D₃ by the exposure of skin to solar UV radiation through a printed strip that acts as a UV molecular dosimeter linked to a software developed to run on a smartphone that converts the UV dose into the amount of vitamin D₃ produced in the skin. The strip is printed using Dimatix DMP2831 MEMS-based Drop-on-Demand materials printer with a photonic functional ink based on Eu(btfa)₃·bipy, which we produced to act as a molecular dosimeter presenting a memory effect by photodegradation. The functional paper strips provide the photonic signal used as input in the software for correlation between UV dose and vitamin D₃ produced in the skin. Since this cutaneous production can provide more than 90% of the daily dose needed by the human organism, we concluded that this solution can contribute to the monitoring of the enhancement of the immune response of individuals since the serum 25-hydroxyvitamin D (25(OH)D) level may be related to its immune response. In addition, the software developed includes the production of a cloud data lake capable of mapping population data on vitamin D deficiency, helping to define public health policies.

Keywords: printable dosimeter, UV molecular dosimetry, vitamin D₃, personal dosimetry.



1. INTRODUCTION

Cutaneous production of vitamin D by exposure of the skin to solar UV radiation can provide more than 90% of the daily dose needed by the human organism and may be the best option to reduce oral supplementation. Especially before completing the vaccination schedule in the COVID-19 pandemic scenario, populations needed to stay indoors longer to avoid contamination, thus reducing sun exposure. In view of this, it should be necessary to restore levels of this vitamin, which also plays an important role in the immune system.

Dror *et al.* (2022) showed that among patients hospitalized with COVID-19, a preexisting vitamin D deficiency in patients infected with the SARS-CoV-2 virus was associated with increased disease severity and mortality through a relationship between pre-infection serum 25-hydroxyvitamin D (25(OH)D) level [1].

To correct vitamin D deficiency in the body, cutaneous production of this vitamin may be the best option to reduce oral intake and can be monitored by personal UV dosimetry since this is the range of solar radiation related to its production. Twenty years ago, our LandFoton Research Group presented at the 13th International Conference on Solid State Dosimetry, the very first molecular nanodevice for personal ultraviolet dosimetry [2]. In that time, these personal dosimeters were produced by Physical vapor deposition (PVD) of a lanthanide complex designed to act as a molecular nanodevice, but with high production cost due to starting material losses, leading to scaling limitations. Recently, a printable version of this UV personal dosimeter allowed scaling [3], and the patent for this printable device was issued this year [4].

In the present work, thanks to a project approved in the Brazilian SibratecNano Program, we present the first results of a solution for monitoring personal vitamin D production status through the use of printed UV dosimeters, using a smartphone for a correlation between received ultraviolet dose and vitamin D production. The active part of the proposed device consists of printed dosimetric strips, as presented at the 1st Latin American Congress on Solid State Dosimetry and Radiation Measurements (LASSD 2021, Recife, Brazil) [5].

2. MATERIALS AND METHODS

The range of the solar spectrum related to vitamin D production is UVB (280-315 nm), which, upon reaching the skin, photoisomerizes 7-dehydrocholesterol (7-DHC) to form pre-cholecalciferol, which is isomerized into cholecalciferol (vitamin D₃).

Aiming to monitor UVB dose looking for correlation with skin production of vitamin D₃, a printed strip with the molecular dosimetry device was developed to produce a photonic signal as input for calculations in software developed here to run on a smartphone.

For this, the molecular dosimeter Eu(btfa)₃·bipy, designed and prepared as reported elsewhere [6,7], was prepared to be printed as a functional ink with a MEMS-based Drop-on-Demand Dimatix DMP2831 Materials Printer (FUJIFILM) to produce the functional paper strips [3].

This piezo-driven drop-on-demand (DoD) material printer uses cartridges DMC-11610, which integrate 16 printheads with 21 μm diameter nozzles coupled with 16 silicon single-crystals (PZT) in micro-electro-mechanical systems (MEMS), jetting drop volume of 10 pL. For higher resolution, it is possible to use 9 μm diameter nozzles jetting drops of 1 pL.

For device printing, an optimized 16 μs electric waveform determines the ramp voltage [3] applied to the piezoelectric actuators (PZT) of the materials printer to eject from the nozzle the droplets carrying the active part of the UV molecular dosimeter, that acts as a Light Conversion Molecular Device (LCMD).

The printable photonic ink is a 0.6 ppm solution of Eu(btfa)₃·bipy, obtained through sonication for 20 mins (180 W, 40 kHz) of 6.0 mg of this active complex dispersed in 9.5 ml of ethanol, followed, after total dispersion, by dropwise addition of 0.5 ml of monoethylene glycol (MEG) under sonication for an additional 10 min. The waveform contains three segments: a 'fill phase' (4 μs at a constant voltage of -12 V), a 'print phase' (6 μs, 17 V), and a 'recovery phase' (3 μs, 8 V). The low average voltages were required to allow the drop formation. This set control algorithm was used in the development of the dosimeter strips.

The DoD printing technique was chosen for the better homogeneity of the molecular films produced, due to a tunable process controlled by a print control algorithm that uses an optimized electrical waveform applied to the piezoelectric actuators to eject the active compound through the printer nozzles monitored by a fiducial camera.

This technique yields a layer-by-layer (LbL) process that enables the production of films with high resolution, leading to the adjustment of the sensitivity of the printed dosimeter as a function of the number of layers. In addition, the DoD technique allows a flexible manufacturing system, and the digital templates ensure the final design and enable the production of the device, together with an easier process and a better low-cost strategy compared to other techniques to produce the printable dosimeter.

All dosimetry measurements were monitored in redundancy with a NIST traceable calibration dosimeter (IL390C Light Bug, SN 4207) from International Light Inc, since a digitally controlled DNA crosslinker is used for UV expositions in all proof-of-concept experiments.

The printed devices were exposed to selected doses of UV radiation in the chosen range, using a Transilluminator UV DNA Crosslinker (UVP Inc.), that uses three sets of five Hg bulbs (5x8 W each one) as UV sources - centered at 254 nm for UVC range, 290 nm to 320 nm for the UVB range and filtered and centered at 365 nm (320 to 400 nm) for UVA range, digitally controlled by a build-in dosimeter, providing 0000.1 - 9999.9 mJ/cm² (0 - 10 J/cm²) exposures. This system is coupled by an optical fiber to a USB4000 spectrometer for higher sensitivity measurements, or a Maya spectrometer for higher resolution (both from Ocean Optics), for the acquisition of real-time luminescence spectra during UV dose exposures.

3. RESULTS AND DISCUSSION

3.1. Printable UV molecular dosimetry

We previously proposed some lanthanide complexes for UV dosimetry, such as presented in the patent claims of the printable UV dosimeter [4]. In the present case, the Eu(btfa)₃·bipy complex was chosen to act as an active part of UV dosimeter to predict personal vitamin D production due to the high-quality fluid obtained for DoD materials printers [3]. The synthesized complex can be seen by itself as a molecular dosimeter: the btfa ligand (4,4,4-trifluoro-1-phenyl-1,3-butanedione) acts as a UV antenna and the bipy ligands (2,2'-bipyridine) shield the Eu³⁺ ions that produce a red luminescence at 612 nm that is inversely proportional to the accumulated UV dose. A

photocleavage mechanism mimics the skin damage, providing a memory effect that enables dosimetry. By using a cellulose-based paper as a substrate to print the active part on a strip for UV dosimetry, the term “Intelligent Paper” can be applied for the printable device in its strip form [3].

The Dimatix Materials Printer used in this work has a built-in camera named “Drop Watcher”, and Figure 1 (a) shows an example of the droplets formation in a stroboscopic image captured by this embedded camera, showing five of 16 nozzles jetting the fluid containing the active complex into the DMC-11610 cartridge, fillable with 1.5 ml. This printable fluid is presented in Figure 1 (b) showing its red luminescence under UV excitation, and Figure 1 (c) shows the printed strip constituted of 20 jetted layers over a cellulose-based paper as a substrate. The observed magenta color is the result of the additive light color synthesis composed of the red luminescence of the Eu^{3+} complex and the blue luminescence of the cellulose-based paper.

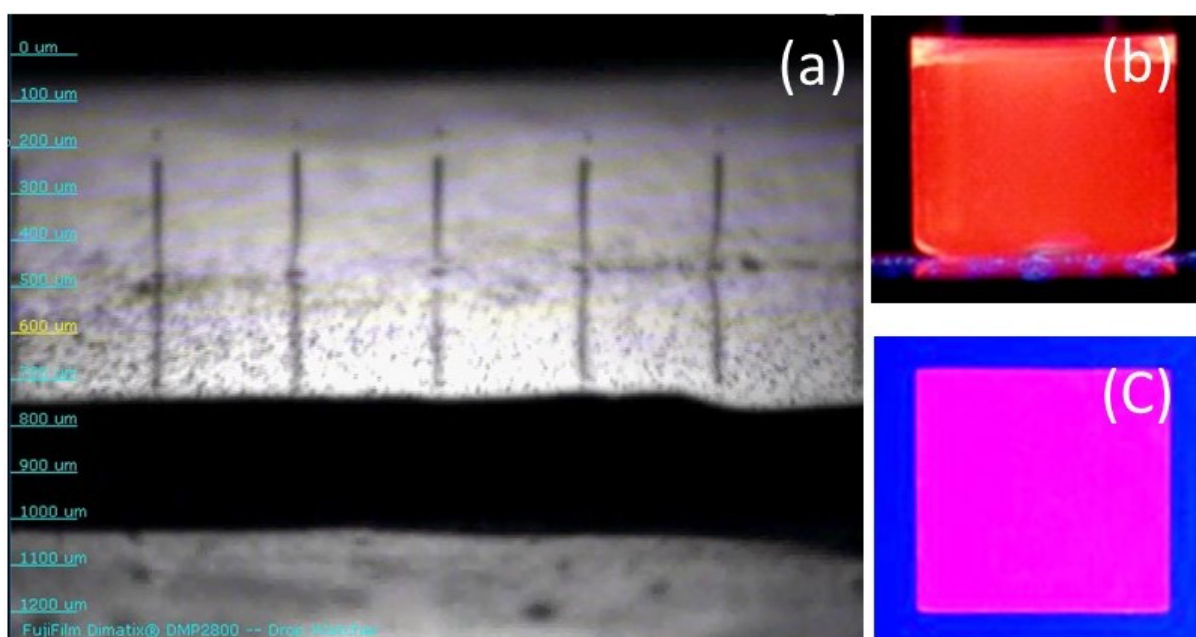


Figure 1: (a) DMC-11610 cartridge jetting the complex in 5 drops of 10 pL from 5 of the 16 nozzles; (b) $\text{Eu}(\text{btfa})_3\text{bipy}$ fluid under UV excitation; (c) printed target under UV excitation: 20 jetted layers over a cellulose-based paper [3].

Figure 2 shows the luminescence spectra of the $\text{Eu}(\text{btfa})_3 \cdot \text{bipy}$ complex used as the active part of the printed UV dosimeter, hereafter simply called the UV dosimeter, as a function of relative UV exposure dose. In this figure, the luminescence spectrum of the unexposed sample is shown as zero dose, and the following spectra refer to exposures at doses up to six times the relative dose 1, to which the material was exposed before taking the spectra.

The spectra show the Eu^{3+} transitions in the visible range, in which the red emission (around 612 nm), related to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition, is the most intense and therefore will be taken for dosimetry purposes [2, 6], to reduce noise in higher cumulated doses (lower luminescence), that may increase the error in the vitamin D production calculations here. Other Eu^{3+} electronic transitions can be observed with their energy levels in our previous work [3] but will not be used here.

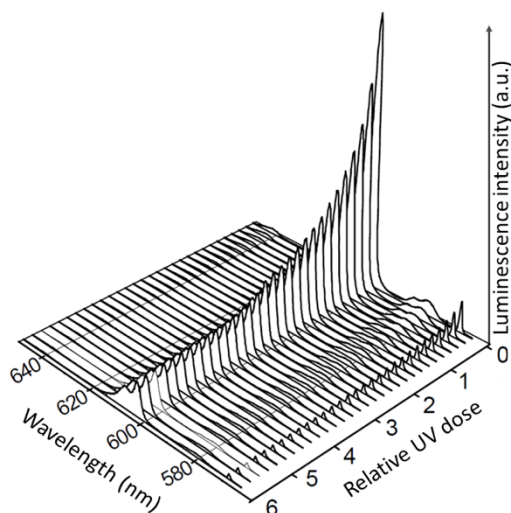


Figure 2: $\text{Eu}(\text{btfa})_3 \cdot \text{bipy}$ spectra showing the Eu^{3+} luminescence quenching as a function of UV relative dose exposure [2].

The printed molecular devices have already been exposed to UVA, UVB, and UVC doses in the range of $0.5\text{--}10 \text{ J/cm}^2$ [3], but due to the skin production mechanism of Vitamin D, the dosimetric calibration curves for UVB (290 nm to 320 nm) were taken as shown in Figure 3, for the printed molecular devices prepared with 20, 30, and 50 jetted layers. To produce the UVB radiation

exposure and also for the spectra recording, as mentioned previously, a UV DNA Crosslinker (UVP Inc.) digitally controlled by a build-in dosimeter was used, also monitored in redundancy with a NIST traceable calibration dosimeter (IL390C Light Bug, SN 4207) from International Light Inc.

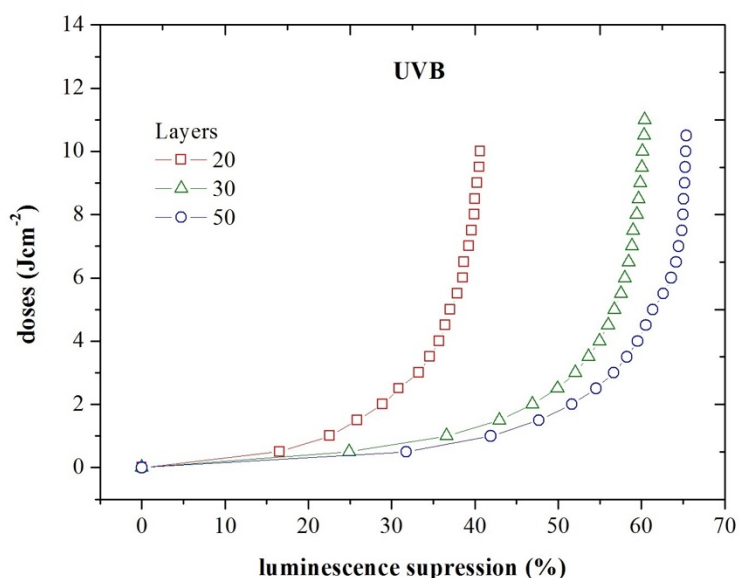


Figure 3: *Dosimetric UVB calibration curves for printed devices prepared with 20, 30 and 50 jetted layers, showing the cumulated dose received as a function of the Eu^{3+} red emission quenching. Source: adapted from [3].*

These dosimetric calibration curves were calculated taking the ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ Eu^{3+} transition intensity at 612 nm, from the luminescence spectra of the printed complex, since this red emission intensity decreases in a well-behaved form as a function of all UV-range of dose exposure. In addition, it was observed that the device sensitivity could be tuned as a function of the number of printed layers. It enables the use of $\text{Eu}(\text{btfa})_3 \cdot \text{bipy}$ as a UV molecular dosimeter by determining the cumulated UV dose received just by measuring its luminescence quenching percentage in J/cm^2 for quantitative dose measurements (Figure 3). The personal UVB dosimetry data can be used as input data to calculate the amount of vitamin D_3 produced during skin exposure.

3.2. Correlation between UVB dose and cutaneous production of vitamin D

Our first concerns that motivated the development of a personal dosimeter for UV radiation were related to the risk of skin cancer [6], so to monitor the cutaneous production of vitamin D based on UV exposure, we must consider a UV Dose Window (UDW) set between two boundaries: 1) an upper limit, given by MED (Minimal Erythema Dose), defined as the lowest dose that caused minimally perceptible erythema for six skin types, related to melanin index, and 2) a lower limit, given by MDD (Minimal vitamin D Dose) the amount needed to produce the recommended daily dose of vitamin D (1000 IU) [8,9].

The dose of UV radiation required to produce the prescribed dose to meet the user's daily requirement should be within this UDW, but it can also be spread over a predetermined period since the vitamin D production depends on UVB dose but not on dose rate [10].

Here, to use our personal UV molecular dosimeter signal as input data to calculate the amount of vitamin D₃ produced during skin exposure, the UV dose obtained by the photonic signal, a correlation curve must be established to convert the UV dose into the amount of vitamin D₃ produced.

Three options are presented for this calculation. As the UV spectrum for MED calculation is different from the UV action spectrum for vitamin D production, we propose to use a UVB filter in the dosimeter to use the first two calculation options, since the vitamin D production efficiency is peaked in this range. As an early option in our App, we propose to use a basic relationship (Equation 1) described by Bogh *et al.* (2010) [10]:

$$\text{PRODUCED VITAMIN D} = 3.5(\text{UVB DOSE}) + 14.3 \quad (1)$$

where UVB DOSE=NUMBER OF SEDs \equiv number of MEDs *PPF

where the number of MEDs= measured UVB dose/1MED(UVB)

in which MED=Minimal Erythema Dose (related to the UV range)

and PPF = pigment protection factor (related to the melanin amount in the skin).

In this relationship, the UVB dose is introduced in number of SED, the Standard Erythemal Dose (1 SED=100 J/m² at 298 nm using CIE erythema action spectrum) and is the MED multiplied by the PPF, the pigment protection factor, related to the melanin amount in the skin. The produced amount of vitamin D obtained by this relationship is related to the increase of serum vitamin D level expressed as 25-hydroxyvitamin D₃ (25(OH)D) in blood samples [10].

Our printed strips cover a wide enough range for this purpose, as they achieve up to 1000 times the MED-UVB, for ~70% of luminescence signal quenching, while showing high resolution around the MED values. Calculations using the relationship between vitamin D production and UVB dose, shown in Equation 1, can be performed by a smartphone App using the printed strip signal as the input value related to the UVB dose, for personal monitoring, taking information about one of the 6 skin types by comparison in a color scale presented in the App.

A more accurate relationship, including more parameters, such as exposed body surface area (BSA), was proposed by Jager *et al.* (2018) [11], taking into account meta-regression results using meta-analysis of observational studies in epidemiological guidelines for the impact of UV dose when the baseline 25(OH)D concentration in blood is known (Equation 2):

$$\text{PRODUCED VITAMIN D} = 0.19(\text{UVB DOSE}) + 30.14 - 0.39 \times (\text{baseline } 25(\text{OH})\text{D}) + 0.05 \times (\text{BSA}) \quad (2)$$

where UVB DOSE is measured in the same way showed in Equation 1, taken in number of SEDs= number of MEDs *PPF (see Eq. 1);

25(OH)D baseline may be estimated or measured in a blood analysis;

And BSA is the exposed body surface area (%), which depends on what clothes the person is wearing, and is also calculated in the App.

By using Equation 2, taking a baseline 25(OH)D serum concentration of 50 nmol/L and the exposure of 10% BSA to a UVB dose of 1 SED, the increase in serum vitamin D level expressed as 25-hydroxyvitamin D₃ (25(OH)D) in blood samples can be estimated to be ~16 nmol/L, meaning that 1 SED can be effective in maintaining a healthy vitamin D status [11]. The BSA calculation tool in our smartphone App allows a more accurate measurement in this calculation option in case of a baseline 25(OH)D data available.

A third option to calculate vitamin D production from the UV exposure dose was presented by Serrano (2018) [9], estimating the amount of daily vitamin D produced from a daily personal median of personal exposure to erythemal UV radiation.

Initially, Serrano converts the daily dose of personal UV erythema exposure (measured UV dose, in J/m^2) to a vitamin D factor dose (UVD, in J/m^2) using climate and geographic corrections that appear as action spectrum conversion factors (ASCF), as shown in Equation 3 [12]:

$$UVD (J/m^2) = \text{measured UV dose } (J/m^2) \cdot \text{ASCF} \quad (3)$$

In this option, UVD dose may be estimated by using the printed dosimeter strip without filters for UVB, acting directly as a broadband personal dosimeter, if the ASCF factor is available for the region.

To calculate by this way the Vitamin D produced, we will need the MDD value, as a newly defined minimum UV dose according to the CIE/WMO (2014) guidelines [13], and Equation 4 calculates the MDD as a function of skin type [14], needed to produce the daily recommended dose of vitamin D (1000 IU):

$$MDD (J/m^2) = (30 J/m^2) / (\text{STF} \cdot \text{BSA}) \quad (4).$$

where BSA is the exposed body surface area (%) as previously defined, which depends on what clothes the person is wearing and is also calculated in the App and STF is the skin type factor.

Given a full-body exposure (Type II skin) under sunlight with $UVI = 10$, under these conditions, the recommended dose of vitamin D would be produced in just one minute [14]. The skin type factor (STF) is used for each skin type: $STF = 250/MED (J/m^2)$, then, to skin type II, where the MED is $250 J/m^2$ [15], STF is 1. The percentage of body exposure (BSA) may be calculated by the aid of a tool embedded in the smartphone App developed here, while STF is calculated by entering the skin type (I-VI), guided by a scale related to the amount of melanin.

Using these data, equation 5 is the last option for the calculation of the amount of vitamin D produced by a person exposed to solar UV radiation:

$$\text{Vitamin D (IU/day)} = (UVD J/m^2/\text{day} \cdot 1000IU) / (MDD J/m^2) \cdot (AF/SPF) \quad (5)$$

where AF is the age factor and SPF is the sun protection factor of any sunscreen applied over the entire exposed body surface. The age factor for children, for instance, $AF = 1$ and for adults between 30 to 50 years old, $AF = 0.7$ [16]. Regarding SPF, two possibilities were considered: unprotected skin ($SPF = 1$) and the use of sunscreen cream ($SPF = 15$ in spring/autumn and $SPF = 30$ in summer) on the entire exposed body surface [9].

3.3. Smartphone App

The smartphone application developed here allows the end-user to monitor their cutaneous vitamin D production by taking as input the photonic signal generated by the printed strip. As shown in Figure 4, the App is being designed in a smart way to estimate the amount of vitamin D produced by the user by combining this signal with user inputs (such as skin coverage, age, etc.), allowing a more accurate correlation between UVB dose and vitamin D₃ production. Fig 4 (a) and (b) shows two screenshots, while Fig 4 (c) shows the prototype device, attached to the top of the smartphone over the luxmeter beside the camera, used to capture the luminescence intensity, inversely proportional to the accumulated UV dose, from the stimulated printed strip for a proof of concept.

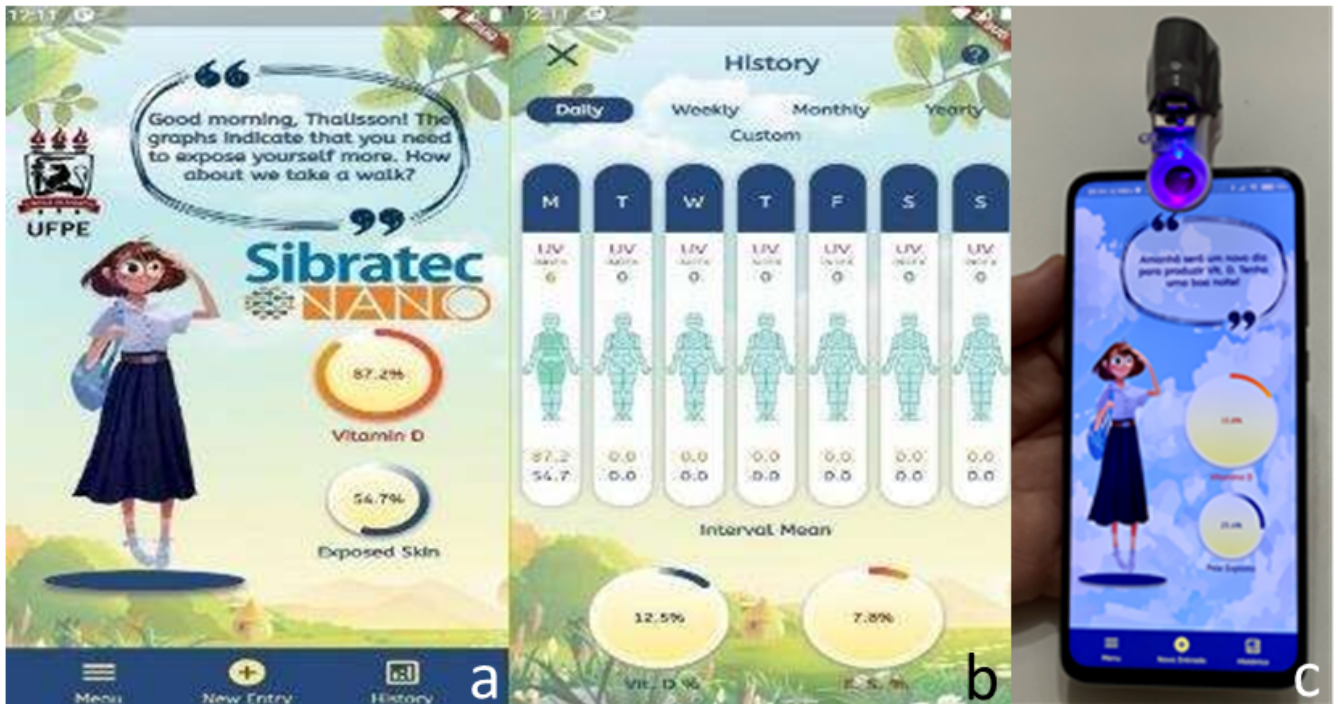


Figure 4 (a) and (b) Screenshots from the App for personal monitoring of cutaneous vitamin D production; (c) Smartphone App with the prototype device attached on top for a proof of concept, showing the luminescence of the printable strip.

The screenshots shown in Figure 5 highlight (a) the initial personal input data, developed to know the user's profile and displayed at registration, including the skin type characteristics, according to Fitzpatrick (1988) [15]; (b) a tool in which the user can select the area covered by the clothes and calculates the amount of exposed skin, in addition, being possible to store frequently wearable clothes to be quickly chosen each day; (c) a history showing the percentage achieved for the vitamin D production goal that was medically prescribed in a safer way (in dark blue) and a visual information in the form of a double bar chart, for a selected custom time window in various time slices, and there is also information about skin health safety related to the ultraviolet index.

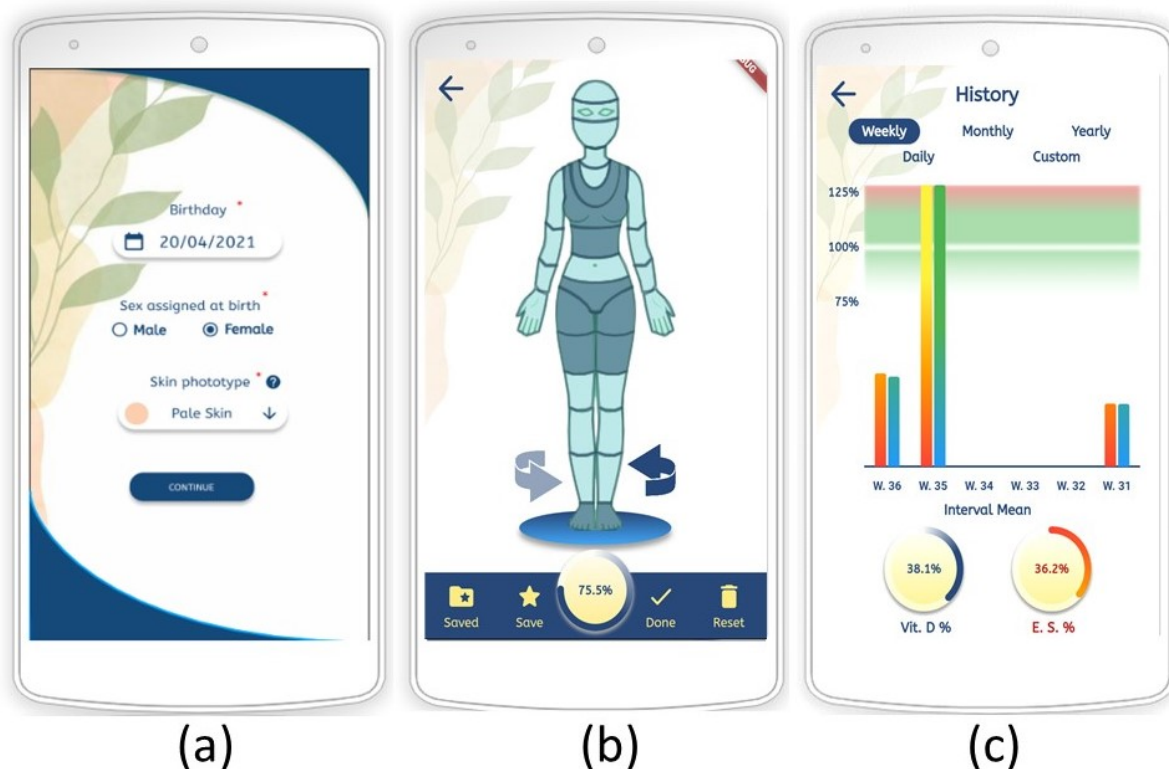


Figure 5 (a) Initial page of the App for personal monitoring of vitamin D production; (b) BSA calculation tool; (c) History page with the estimated cutaneous production of vitamin D.

Although the UV dosimetry data has been previously validated for the printable dosimeter [3,4] and the App presented here provides the conversion of the UV dose into an estimated personal production of vitamin D, according to the proof of concept showing the feasibility, it is still necessary to validate the system through a comparative analysis of the conversion options presented here. Thus, this is a potential solution for low-cost personal monitoring of the skin production of vitamin D with an option for oral supplementation, aiming to contribute to a better immune response of individuals.

4. CONCLUSIONS

We present a promising solution for personal monitoring of cutaneous vitamin D production using printable UV dosimetric strips. A smartphone software has been developed to receive data from the printable strips and information from users, including the percentage of exposed skin, providing as daily information, the percentage achieved to reach the medically prescribed vitamin D dose level. The creation of a cloud data lake to map population data on vitamin D deficiency is planned for the system, which could help to define public health policies.

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