

Laboratory Stand for the Optimisation of the UV Curing of Fluids Disposed on Textiles

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Abstract

Processes of linking polymer coatings which functionalise textiles in the presence of photoinitiators require using high-energy UV radiation sources. However, the values of radiation intensities and UV doses used should be controlled so that they will not be greater than required by the proper course of the linking process, on account of the high degrading impact of UV radiation on the fibre structure. For this reason, optimisation of the radiation process is necessary to ensure the appropriate durability of the coating in conditions of use and when washing the product, thereby minimising fibre degradation. In this article, a laboratory stand is presented for optimisation of the process of the UV curing of polymer coatings that functionalise textiles. A method of calibrating the radiation system is presented in which the programming of parameters can be performed each time without the need to use a system of measurement of the intensity and UV radiation dose.

Key words: disposed fluids, UV curing laboratory stand, UV curing optimisation.

Introduction

The process of linking polymer coatings by means of a UV light source requires providing an optimal dose of UV radiation. As a rule, a UV radiation dose during the linking process ranges from 0.5 to 10 J/cm². On the one hand, increasing the UV radiation dose affects the durability of coatings in conditions of product use, while on the other, an excessive UV radiation dose is economically unjustified and harmful due to its strong degrading effect on the fibre structure. Consequently, this leads to the impairment of the durability properties of a printed textile product. UV radiation doses of an order of 0.5 to 10 J/cm² have a degrading effect on nearly all kinds of fibres used for manufacturing textile products by impairing their durability properties. Mechanisms of photodegradation caused by UV radiation in the case of natural and artificial polymer materials are extensively discussed in publications [1 - 3]. In addition, the high temperature generated in the radiation zone due to the considerable amount of heat given off by the UV source can cause the excessive evaporation of some constituent substances of

the ink being deposited. For this reason, designing an industrial radiation system requires performing a number of experiments on a laboratory stand to optimally select the following [4]:

- the power of the UV source and the geometry of the reflector,
- the distance of the source with a reflector from the surface being irradiated,
- the time of radiation in static conditions,
- the speed of movement of the material in dynamic conditions,
- the temperature in the radiation zone.

In addition to studies related to designing industrial UV radiation systems, laboratory stands equipped with a standard sensor of UV radiation intensity are used for the calibration of UV dosimeters deposited onto textiles, changing its colour depending on the UV dose magnitude. This calibration process consists in finding a functional relationship between the change in dosimeter colour and the UV radiation dose determined by integration according to the UV radiation intensity in a time domain obtained by means of a standard UV sensor. Changes in colour

are determined by measuring the coefficient of remission of light from the active dosimeter surface [5].

The main aim of the investigation was to construct a laboratory stand for optimisation of the processes of the UV curing of coatings e.g. antibacterial and antistatic coatings, which functionalise textiles.

Investigation stand

To optimise the radiation system parameters, a laboratory stand for the linking of functionalising coatings deposited on textile samples using a high-energy UV radiation source was constructed. The following assumptions were made in the construction design development phase:

- the maximum size of the sample irradiated – 20 cm × 30 cm,
- the UV radiation dose – up to 10 J/cm²,
- controlling the UV radiation dose without the need to repeatedly measure its value,
- the possibility of measurement of the temperature in the radiation zone within the range of 10 to 300 °C.

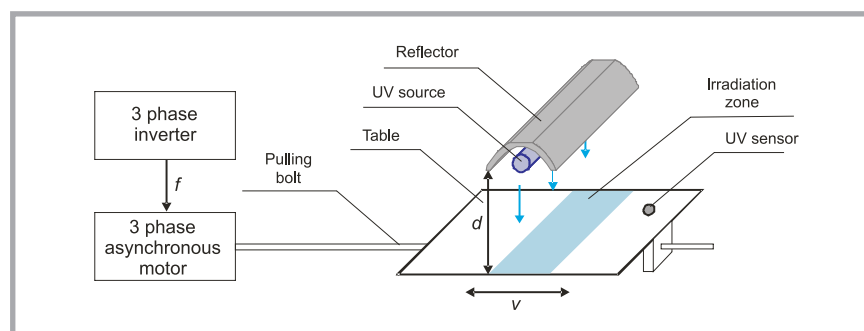


Figure 1. Block diagram of the stand for the irradiation of fabric samples with a functionalising coating.

In **Figure 1** presents a block diagram of the laboratory stand developed for the irradiation of fabric samples with a functionalising coating .

The main elements of the stand are a high-pressure mercury lamp of 2100 W, type HOK 20/100 of Philips make, emitting UV radiation, and a sliding table. A high voltage, medium pressure, mercury discharge lamp is the most popular light source for the UV curing of installations. Lamps in excess of 1m width are available, offering power levels of 80 - 120 W/cm. Over the history of UV curing, both the available power and product life of commercial lamps have gradually increased. The spectrum distribution of the UV radiation of a HOK 20/100 lamp is shown in **Figure 2**. The lamp is placed in a reflector, whose task is to distribute the entire UV radiation over the work surface of the sliding table. The sample, placed on the work surface of the table, is then irradiated by shifting it through the radiation zone. The shift of the table is effected by a nut-bolt system, where the nut is fastened permanently to the table, while the pulling bolt is driven by means of a three-phase asynchronous motor controlled by a frequency converter.

In this system, the UV radiation intensity and dose depend on the parameters of the UV radiation source, the distance d of the source from the sample and the speed v

of the table shift. Assuming constant parameters of the UV radiation source and reflector, the UV radiation intensity and dose during the irradiation of the sample s will depend only on the distance of the source from the sample and the speed of the table shift . Thus, we obtain

$$\begin{aligned} E &= f(d) \\ Q &= f(d, v) \end{aligned} \quad (1)$$

where:

E - the UV radiation intensity,

Q - the UV radiation dose,

While for the time $t \in [t_1, t_2]$, the following relationship holds true [5]:

$$Q = \int_{t_1}^{t_2} E(t) dt . \quad (2)$$

The speed v of the table movement depends on the pitch h of the pulling bolt thread and on the rotational speed n of the asynchronous motor, according to the relationship

$$v = n \cdot h . \quad (3)$$

The rotational speed of the asynchronous motor depends on the frequency f of the supply voltage, as well as on the slip and number of pairs p of the poles. Neglecting the slip due to a small load moment, we obtain

$$v = \frac{60 \cdot f}{p} \cdot h . \quad (4)$$

Given the constant number of pole pairs and constant pitch of the pulling bolt,

the speed of table shift is controlled by means of the frequency of the asynchronous motor supply voltage. Thus, the intensity and dose of UV radiation during the irradiation of sample s can be programmed using the distance d of the UV source from the sample and the frequency f of the asynchronous motor supply voltage. Hence,

$$\begin{aligned} E &= f(d) \\ Q &= f(d, f) \end{aligned} \quad (5)$$

To program the UV radiation intensity and dose, a system was developed for the measurement of these two quantities based on a semiconductor diode with a non-zero sensitivity within a wavelength range of only 200 ÷ 400 nm. At present, no semiconductor sensors for high UV radiation intensities of 5 - 10 W/cm² are produced; therefore, it was proposed that a UV filter reducing the intensity to levels processed by the sensor be incorporated into the optical signal processing circuit.

■ Irradiation system calibration

Using the irradiation process with a high-energy UV radiation source is accompanied by a high increase in the temperature of the exposition zone. The temperature values that can occur reach an order of 150 ÷ 200 °C, as a result of which the continuous operation of the system for UV radiation intensity and dose measurement is not possible. To programme the UV radiation intensity and dose without the necessity of using a measuring system, calibration was performed by determining their functional dependence on the distance d of the source from the table and on the frequency f of the asynchronous motor supply voltage, according to formula (5).

In the first stage, measurements of radiation intensity were made using a constant supply voltage frequency of $f = 15$ Hz, whereas the distance d of the source from the specimen surface was varied from 5 cm to 25 cm, every 5 cm. Investigation results are shown in **Figure 3**.

In the second stage, for the curves $E(t)$ obtained, calculation of the UV radiation dose was made using relationship (2).

Analysis of regression was used for the results obtained. The following functional dependences were found:

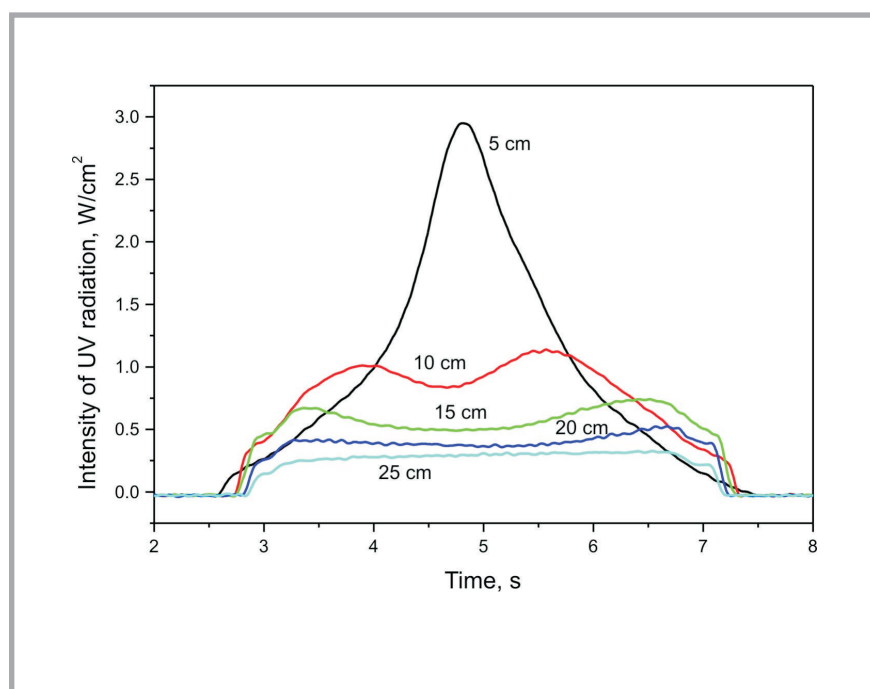


Figure 2. Spectrum distribution of a HOK 20/100 lamp of Philips make [9].

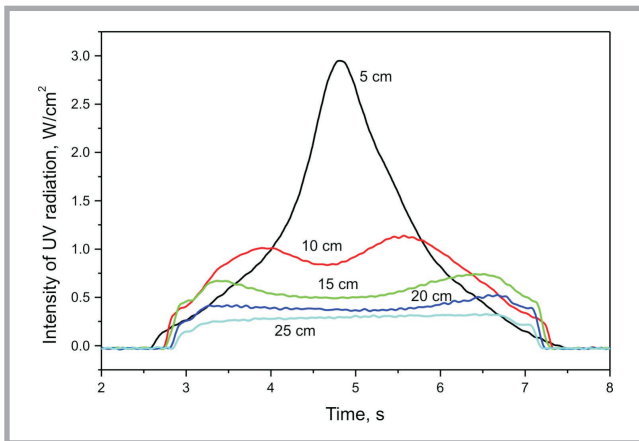


Figure 3. UV radiation intensity as a function of time for different distances of the lamp from the sample surface.

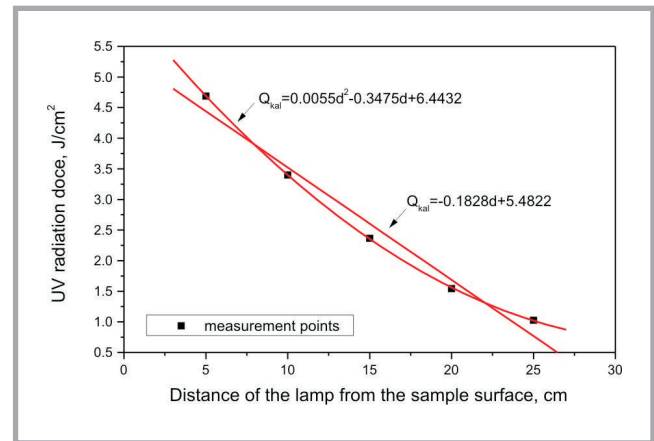


Figure 4. UV radiation dose as a function of the distance of the lamp from the sample surface.

- $Q_{kal} = -0.1826d + 5.4822$ in the case of the linear regression used, with the coefficient of determination $R^2 = 0.9674$, and the function of loss is 1.0650.

and

- $Q_{kal} = 0.0055d^2 - 0.3475d + 6.4432$ in the case of the non-linear regression used, with the coefficient of determination $R^2 = 0.9997$, and the function of loss is 0.0095.

The above dependences between the UV radiation dose and the distance of the light source from the sample surface are shown in **Figure 4**.

Since the UV radiation dose per sample surface unit is inversely proportional to the change in table shift speed, its programming – without the need to make a measurement – can be performed by selecting parameters according to the following formula:

$$Q = \frac{v_{kal}}{v} Q_{kal}(d), \quad (6)$$

where:

- v_{kal} - the linear speed of the table in conditions of determination of the function,
- $Q_{kal}(d)$ - the radiation dose as a function of the distance from the table obtained in the conditions of determination of the function,
- v - the selected linear speed of the table,
- d - the distance of the lamp from the table.

When a frequency converter is used in the system for control of the linear speed of the table, it is more convenient to pro-

gramme the UV radiation dose by selecting the frequency of the voltage of the three-phase asynchronous motor. Thus, we have

$$Q = \frac{f_{kal}}{f} Q_{kal}(d), \quad (7)$$

where:

- f_{kal} - the three-phase voltage frequency in the calibration conditions,
- f - the three-phase voltage frequency selected.

One of the approximating functions, linear or polynomial of the second degree, should be chosen as the function $Q_{kal}(d)$. By selecting the function in the form of a polynomial of the second degree to pre-

cisely programme the radiation dose, we the final formula of the form:

$$Q = \frac{f_{kal}}{f} (0.0055d^2 - 0.3475d + 6.4432). \quad (8)$$

Thus, the definite dose of radiation Q is obtained by selecting the distance d and frequency f . For the investigation stand developed, both parameters can be changed in the following range:

- the distance d - from 5 cm to 25 cm,
- the frequency of the three-phase voltage f - from 5 Hz to 60 Hz.

In **Figure 5**, the dependence of the UV radiation dose on the distance of the lamp from the sample and on the three-phase

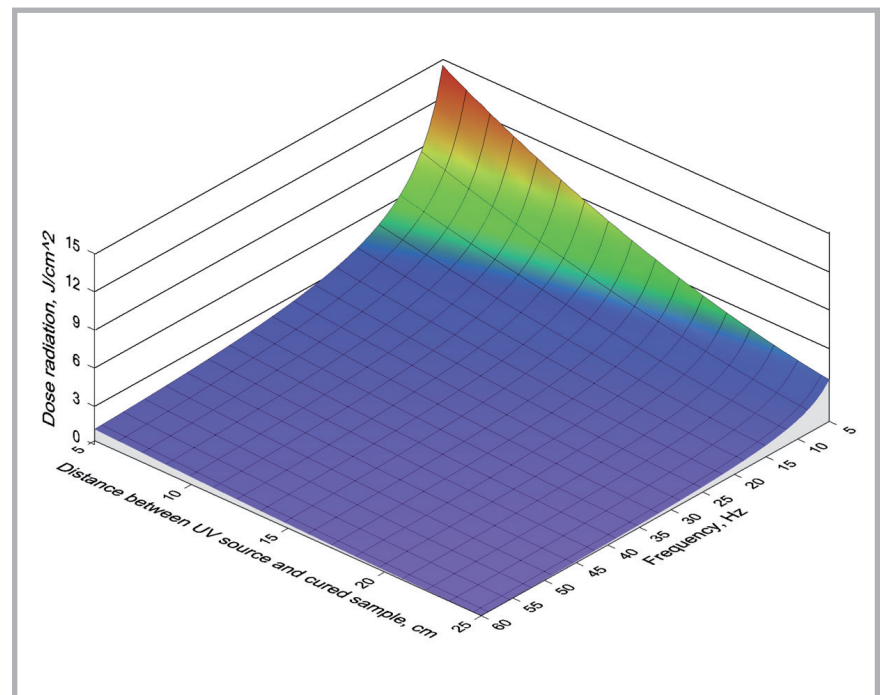


Figure 5. UV radiation dose as a function of the distance of the lamp from the sample surface and the control voltage frequency.

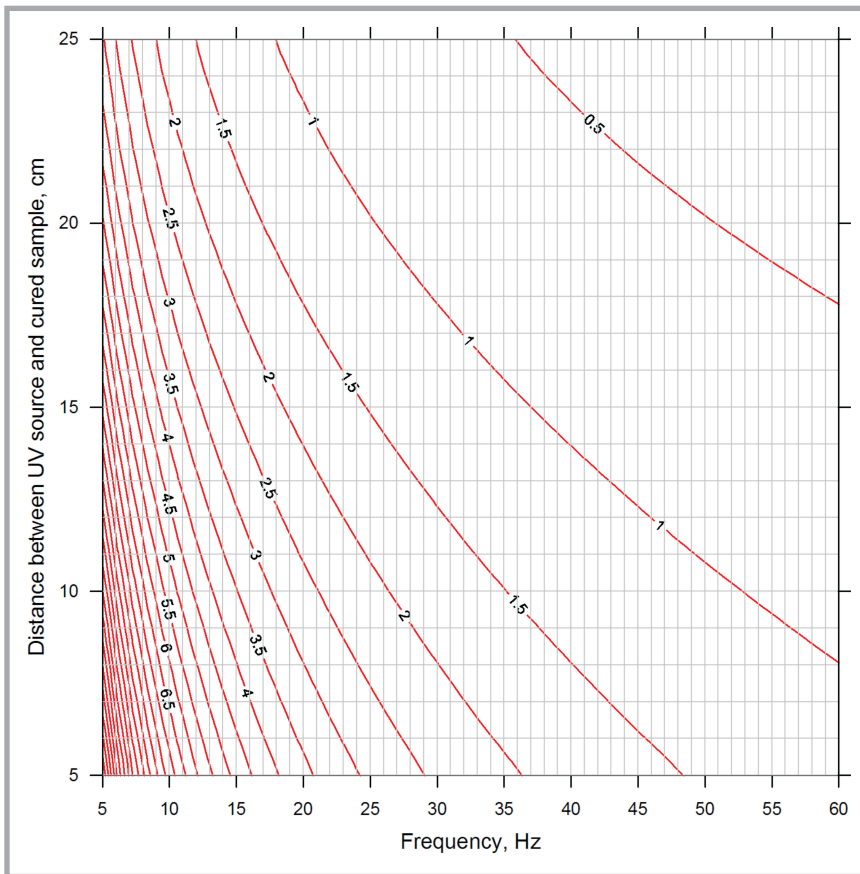


Figure 6. Nomogram for the selection of the value of parameters d and f for a specific UV radiation dose, given in J/cm^2 .

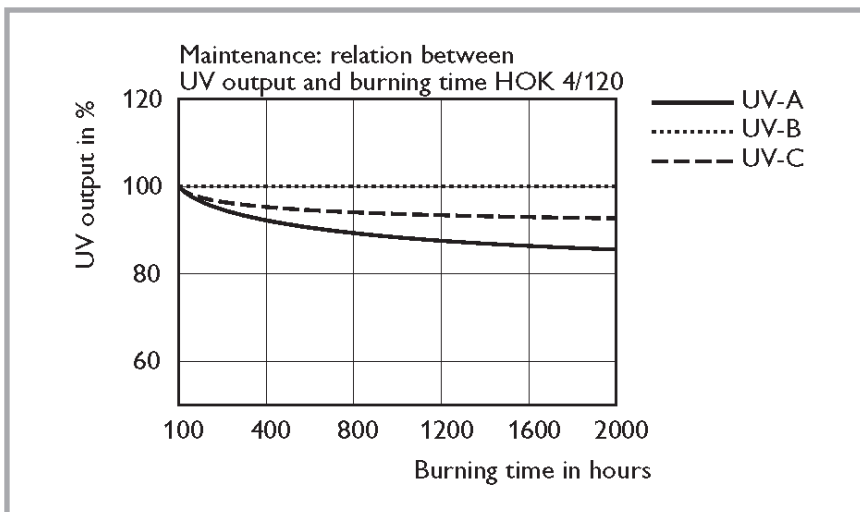


Figure 7. Relation between the UV output and burning time of Philips HOK type lamps [9].

voltage frequency is presented, obtained on the basis of formula (8).

In research practice the value of distance d is selected as high as possible, allowing

- to limit UV radiation affecting the sample,
- to limit the temperature of the irradiation zone.

By projecting isolines of the UV radiation dose, shown in Figure 5, onto the surface using the d - f coordinate system, a very convenient nomogram is obtained (Figure 6) for the selection of the distance of the source from the sample surface and frequency of the asynchronous motor supply voltage so that a definite dose of UV radiation can be determined.

The dependence of the dose of UV radiation Q on the distance d and frequency f determined by formula (8) does not take into account changes in the intensity of the UV radiation of the lamp over time (Figure 7).

In the case of Philips HOK type lamps, the intensity of UV-B radiation is constant, whereas the intensity of UV-C and UV-A radiation decreases, respectively, by about 7% and 14% during the whole product life. As a general rule, in processes of the UV curing of fluids disposed on textiles, small changes in UV dose radiation over time are acceptable. For the laboratory stand presented, the calibration procedure should be repeated every $100 \div 200$ work-hours of the lamp.

Assessment of the inaccuracy of UV radiation dose selection

Analysis of the inaccuracy of the determination of the dose Q of UV radiation of 200 ÷ 400 nm wavelength was carried out on the basis of the uncertainty theory [6, 7]. The complex uncertainty, defined by the formula below, was taken as a measure of the inaccuracy of the dose measurement:

$$U(Q) = k \times u_C(Q), \quad (9)$$

where:

k - the coefficient of expansion ($k = 2$ for a confidence level of 0.95),

$u_C(Q)$ - the combined standard uncertainty of the estimate Q .

The general form of the equation from which the estimate Q of the value of the real UV radiation dose is calculated, resulting from relationship (8), is given by the formula

$$Q = \frac{f_{kal}}{f} (a_2 d^2 + a_1 d + a_0), \quad (10)$$

where:

a_2, a_1, a_0 - the coefficients of non-linear regression of the function

$$f_{kal} = \text{const.} (f_{kal} = 15 \text{ Hz}).$$

Making use of the uncertainty propagation law [6], assuming that the estimates of the input quantities and their errors are modelled by independent random variables, and from formula (10), a dependence is obtained of the combined uncertainty $u_C(Q)$ of the estimate Q from the components of standard uncertainty: f, d, a_2, a_1, a_0 . This relationship has the form

Table 1. Uncertainty budget.

Symbol of input quantity	Estimate of input quantity value	Standard uncertainty of		Variance of input quantity	Sensitivity coefficient square	Value of the component of combined variance of output quantity, (J/cm ²) ²
		Type A	Type B			
<i>f</i>	18.5 Hz	0 Hz	0.1068 Hz	1.1×10 ⁻² Hz ²	0.0117 (J/Hz×cm) ²	1.3×10 ⁻⁴
<i>d</i>	15.0 cm	0.0071 cm	0.0009 cm	5.1×10 ⁻⁵ cm ²	0.0220 (J/cm ²) ²	1.1×10 ⁻⁶
<i>a</i> ₂	0.0055 J/cm ⁴	0 J/cm ⁴	0.0001 J/cm ⁴	1.6×10 ⁻⁸ (J/cm ⁴) ²	33281 cm ⁶	5.3×10 ⁻⁴
<i>a</i> ₁	-0.3475 J/cm ³	0 J/cm ³	0.0039 J/cm ³	1.5×10 ⁻⁵ (J/cm ³) ²	147.92 cm ⁴	2.2×10 ⁻³
<i>a</i> ₀	6.4432 J/cm ²	0 J/cm ²	0.0253 J/cm ²	6.4×10 ⁻⁴ (J/cm ²) ²	0.6574 cm ²	4.2×10 ⁻⁴

$$u_c^2(Q) = \left(\frac{\partial Q}{\partial f}\right)^2 u^2(f) + \left(\frac{\partial Q}{\partial d}\right)^2 u^2(d) + \left(\frac{\partial Q}{\partial a_2}\right)^2 u^2(a_2) + \left(\frac{\partial Q}{\partial a_1}\right)^2 u^2(a_1) + \left(\frac{\partial Q}{\partial a_0}\right)^2 u^2(a_0) \quad (11)$$

An analysis of the inaccuracy of the measurement of the dose *Q* of UV radiation was performed at six points of the nomogram (Figure 7). Table 1 presents an exemplary budget of the uncertainty of the determination of the dose *Q* of UV radiation, assuming that *f*=18.5 Hz and *d*=15.0 cm. Calculations of the standard uncertainties by the B type method were made with an assumption of uniform distribution of possible values within the interval.

Table 2 presents a juxtaposition of the combined, complex and relative uncertainties of the measurement result of the dose *Q* of UV radiation for the six points of the nomogram selected.

It results from Table 2 that the relative uncertainty calculated at the calibration time of selection of the dose *Q* of UV radiation ranges from 3 to 11%. The estimate of variance of the coefficient *a*₁ of the non-linear regression of the function *Q*_{kal}(*d*) in formula (8) has the greatest percentage proportion in the combined variance. Percentage proportions determined for the six points of the nomogram selected range from 55 to 67%. This fact causes a distinct increase in the relative

inaccuracy of the selection of the dose *Q* of UV radiation along with an increase in the distance *d*. The percentage proportions in the combined variance of the remaining estimates of variance of regression coefficients *a*₂ and *a*₀ do not exceed 28%. In the case of the estimate of variance of the frequency *f*, the proportion is below 17%, while in the case of distance *d*, it is below 0.05%.

Conclusions

The laboratory system of irradiation presented allows the optimal selection of the parameters of the industrial system of the UV irradiation of functionalising liquids deposited on textiles as well as the calibration of textile sensors of the UV radiation dose. The durability of the coating in conditions of using and washing the product and the minimisation of fibre degradation were taken as optimisation criteria. The programming of the UV dose during the irradiation process and the calibration of sensors are performed by the selection of the distance *d* of the source from the table and the frequency *f* of the asynchronous motor supply voltage, without repeated measurement of the intensity *E* and dose of UV radiation *Q*.

Using the nomogram developed for the selection of the dose of UV radiation *Q*, one should consider the increasing relative uncertainty of measurement, from 3 to 11%, that occurs in conjunction with an increase in the distance of the lamp from the table.

When selecting the dose of UV radiation *Q*, one should remember about the large increase in UV radiation intensity when the distance of the UV source from the surface is smaller than 10 cm, which can cause the degradation of the fibre structure.

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Table 2. Juxtaposition of the uncertainty of UV radiation dose *Q*.

Points of nomogram		Estimate of output quantity <i>Q</i> , J/cm ²	Combined uncertainty square <i>u</i> ² _c (<i>Q</i>), (J/cm ²) ²	Complex uncertainty <i>U</i> (<i>Q</i>), J/cm ²	Relative complex uncertainty <i>U</i> _{rel} (<i>Q</i>), %
<i>f</i> , Hz	<i>d</i> , cm				
26.40	10.0	2.00	8.2·10 ⁻⁴	0.03	3
18.50	15.0	2.00	3.3·10 ⁻³	0.06	6
12.70	20.0	2.00	1.3·10 ⁻²	0.11	11
53.00	10.0	1.00	2.2·10 ⁻⁴	0.01	3
37.10	15.0	1.00	8.2·10 ⁻⁴	0.03	6
25.40	20.0	1.00	3.2·10 ⁻³	0.06	11