

SPECTROKINETIC INVESTIGATION OF THE PHOTOCHROMIC SYSTEM UNDER CONTINUOUS UV IRRADIANCE USING REFLECTANCE VS. TIME CURVES

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ABSTRACT

The photochromic properties of the dye 5-chloro-1,3,3-trimethylspiro[indoline-2,3'-[3H] naphtho[2,1-b] [1,4]-oxazine] were investigated by exposing it to continuous monochromatic irradiation using 360 nm ultraviolet irradiation using photochromic prints at 293.15 ± 2 K; it undergoes a photocolouration and photodecolouration state, thus following the dynamics of a photochromic cycle of growth and decay phase, respectively. Even if the isolated photo isomeric form of the photochromic dye structure is unavailable, one can quantitatively analyse the photo-induced kinetics of photochromic systems using the raw reflectance data. The quantum colour yield is represented by colour span values [K/S] obtained by converting reflectance data to Kubelka-Munk values. The dynamics of the photoreaction in a photochromic system can be examined by analysing the constants for the rate of colouration and decolouration caused by light. By employing first-order kinetics, the dependent dominant wavelength [λ_{max}] can be determined by fitting the raw data obtained as a change in reflectance values from the FOTOCHROM3 spectrophotometer. The relationship between the colour span values and the concentration of the photochromic dye employed in the photochromic prints is linear.

KEYWORDS

Photokinetics; Photochromic system; Spiroxazine dye; Colour span values; UV irradiance.

INTRODUCTION

Photochromism is a photoinduced reversible phenomenon, as it undergoes colour transformation upon UV irradiance, and its removal goes to its colourless form. Spiro-[indoline-naphthoxazines] and spironaphthoxazines are two well-known photochromic compounds. They comprise two heterocyclic moieties linked by a tetrahedral spiro-carbon to stop conjugation. Figure 1 shows how the spironaphthoxazine-based photochromic dye changes colour when exposed to light. The dye is 5-chloro-1,3,3-trimethylspiro [indoline-2,3'-[3H] naphtho[2,1-b] [1,4]-oxazine].

When light hits the spiroxazine-based chromic dye, the spiro-carbon (C_{spiro}) bond breaks apart. Thermal cis-trans isomerisation into the zwitterionic or quinonoid merocyanine structure follows this process. If you change the amount of UV light that a certain

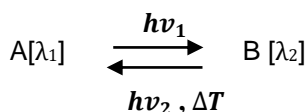


Figure 1. Photochromic phenomena and its interconversion between two states.

type of chromophore absorbs and the number of molecules that make up that light, the molecules change at the molecular level and behave as H-aggregates and J-aggregates. This causes the red and blue shift in the electromagnetic spectrum from its main wavelength. The colourful transformation over the measured UV irradiance cycle is considered a change in energy level between its ground and excited states. This process is crucial for understanding the molecular structure's reactivity, stability, and physical transformation in its pure form. However, the screen-printing method micro-encapsulates the photochromic dye, making it less pure, and prepares the printing paste with additional additives. The stability of these photochromic systems under UV light, the duration of their exposure, and their lack of colour influence their reaction to UV light. These things influence how they react backwards when exposed to visible light [1-3].

Cotton-based photochromic fabrics use technological methods to develop a photoresponsive system, including printing, coating, padding, sol-gel, and exhaust [3]. Some technological procedures for producing photochromic systems include mass dyeing, melt spinning, screen printing, inkjet printing,

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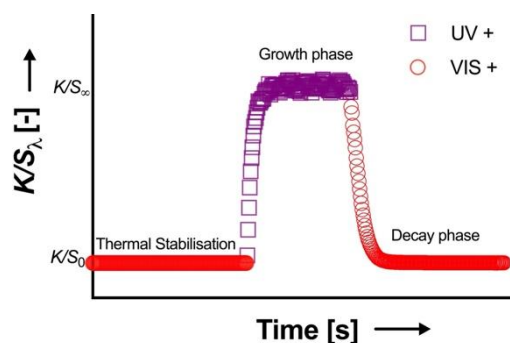


Figure 2. General kinetic profile curve of a photochromic cycle.

pad-dry-cure, exhaustion dyeing, and electrospinning. Each technical method offers distinct merits and drawbacks. Little et al. prepare the photochromic fabric using screen printing and photochromic colourants as dispersed dyes [4,5]. Viková et al. constructed the chromic system using a variety of photochromic prints and studied colorimetric properties using the screen-printing method [6–9]. Solanki et al. developed the photo-fatigue measurement methodology, which irradiates the photochromic system under UV irradiance (the exposure time in seconds) and decolourisation time. This results in the upper plateau of the typical curve of a photochromic cycle for photochromic prints made using screen-printing with the same photochromic dye [8, 9].

The dynamics of kinetic behaviour in a photochromic cycle can be illustrated in Figure 2. The photochromic cycle curve illustrates the colouration and decoloration modes of conduct when a UV light source is present and absent, respectively, in a continuous mode. It starts with the thermal stabilisation (VIS+), followed by the growth phase (UV+) and the decay phase (VIS+) [6-9].

Several studies provide valuable insights for modelling the kinetics of a photochromic system in continuous mode. Pimienta et al. [10] investigate the kinetics of spiropyran photochromic systems under continuous light exposure. This allows us to look into the kinetics of photochromic compounds in solution. Bouas-Laurent & Dürr [11] discuss fatigue, main photochromic parameters, and spectrokinetic aspects, all of which are critical for understanding the behaviour of photochromic systems under continuous irradiation. Maafi et al. [12] present useful spectrokinetic methods for investigating photochromic kinetics, offering formalisms and procedures to analyse photochromic system kinetics. We can learn a lot about how to model the kinetics of photochromic systems in continuous mode by looking at things like fatigue, spectrokinetic methods, structural influences, and different ways to test and characterise the kinetics of photochromic systems. The photokinetics of the photochromic pigment under continuous irradiation vary from one state to another.

However, the essential parameters considered when conducting such studies are the following:

1. Dependence of the absorption of UV irradiance
2. Choice of media or substrate, the polarity of the base material
3. Choices of light sources of a specific wavelength
4. Choice of concentration, following Lambert's law
5. Monitoring the reaction
6. The reaction procedure is temperature control during the measurement, leading to the backward reaction and thus adding photodegradation due to the temperature.

Photo-induced kinetic modelling is essential for understanding the behaviour of photoreversible photochromic systems, which undergo colouration (growth phase) and decolouration (decay phase). The spectral absorbance value (measured with a spectrophotometer), the quantum yield (colouration value), the temperature (measurements were done at room temperature), the concentration of the photochromic dye, and the base substrate can be a solvent form that is applied to the surface of the fabric or doping in the form of a polymeric matrix. All of these things can change the kinetics of photochromic compounds. These factors impact the photochemical and thermal reaction kinetics, which are crucial for predicting the colour performance of photochromic textiles [13, 14]. To understand the photokinetics of photochromic prints, it is essential to consider the complex relationship between photochromic compounds and the kinetic processes governing their colouration and decolouration reactions. Seipel et al. [13, 14] introduced an extended kinetic model that defines rate constants for colouration, decay, and decolouration, allowing for predicting colour performance in photochromic textiles. This model offers a thorough comprehension of how specific fabrication parameters, like inkjet printing and UV-curing processes, can modify the kinetics of the photochromic reaction. We commonly use first-order kinetics models to accurately describe photochromic colour behaviour, representing colouration and decolouration phases with their respective rate constants [17]. Kinetic modelling and its parameters can elucidate the complex interplay of kinetic processes in photochromic prints. By studying the kinetic behaviour of photochromic dye as photochromic prints, researchers can uncover new opportunities for designing smart textiles, optical devices, and functional materials.

EXPERIMENTAL

Photochromic prints preparation

Six photochromic systems were created using different concentrations of a 5-chloro-1,3,3-trimethylspiro[indoline-2,3'-[3H] naphtho[2,1-b] [1,4]-oxazine] dye: 50 g.kg⁻¹ (Purple A), 100 g.kg⁻¹ (Purple

B), 150 g.kg⁻¹ (Purple C), 200 g.kg⁻¹ (Purple D), 250 g.kg⁻¹ (Purple E), and 300 g.kg⁻¹ (Purple F). Figure 1 displays the utilised photochromic dye's closed and open forms.

Kinetic measurements using a FOTOCHROM3 spectrophotometer

The spectrophotometer FOTOCHROM3 (Figure 3(a)) was used to perform photo-induced kinetic measurements to assess the photochromic system's resistance to fatigue induced by continuous UV illumination. The gadget uses the CHROMADYNAMIK software to give real-time colour measurements in UV and visible light sources and deliver continuous irradiance mode spectrum reflectance data. The samples were subjected to continuous and cyclical exposure to both light sources using a measuring aperture with a diameter of 28 mm. The photochromic prints were subjected to a continuous UV LED light with a 360 nm wavelength and a spectrum irradiance strength of 799 W.cm⁻². This radiation source was used to assess the photo colouration of the chromic systems. The FOTOCHROM3 device enables the measurement of a photochromic cycle's UV+ and VIS+ phases. It uses a 360 nm UV light source (UV+) and a visible light source (VIS+) to measure the kinetics of the photochromic system. This allows for measuring changes in reflectance data over time during the photo colouration and photodecolouration phases. Figure 3(b) shows the power distribution of the UV LED and visible light sources used in the spectrophotometer, as measured by a spectroradiometer.

The experiment's environmental parameters included an ambient relative humidity of 45–55 % and a

measurement device temperature of 20°C ± 2°C. The UV irradiance dose was measured as UV dose [mW.s.cm⁻²] = UV energy received [mW.cm⁻²] * UV irradiance time [s] used for UV exposure under continuous UV irradiance using the FOTOCHROM 3 device. The photo colouration UV irradiance time was 240 s, and the photo decolouration (photobleaching) time was 360 s for all photochromic systems.

Data treatment

The first-order association model describes the photo colouration transformation reaction process when the photochromic system is exposed to UV irradiance. The first-order dissociation model (Figure 4(a)) represents the photo colouration of colour intensity. Upon removing the UV light source, the chromic system tends to regain its original form under the fluence of the used visible light source; the relaxation phase follows the first-order dissociation model (Figure 4(b)). Under continuous monochromatic irradiation, the photochromic system can reach its photo-steady equilibrium state (the upper plateau curve of the growth phase) and in opened (coloured state) form. Under continuous UV irradiation, we used a base substrate as a textile fabric form. This colour change during the photo colouration phase is measured as a change in the reflectance, absorbance, or scattering, which can be visualised as chromogenic.

When exposed to a UV light source, the photochromic dye's colour change is quantified using the Kubelka-Munk function. The Kubelka-Munk function, along with Beer's law, is characterised by having a single wavelength. The colour values (K/S) were determined using the Kubelka-Munk function, equation 1 [8,9], to the reflectance spectral data.

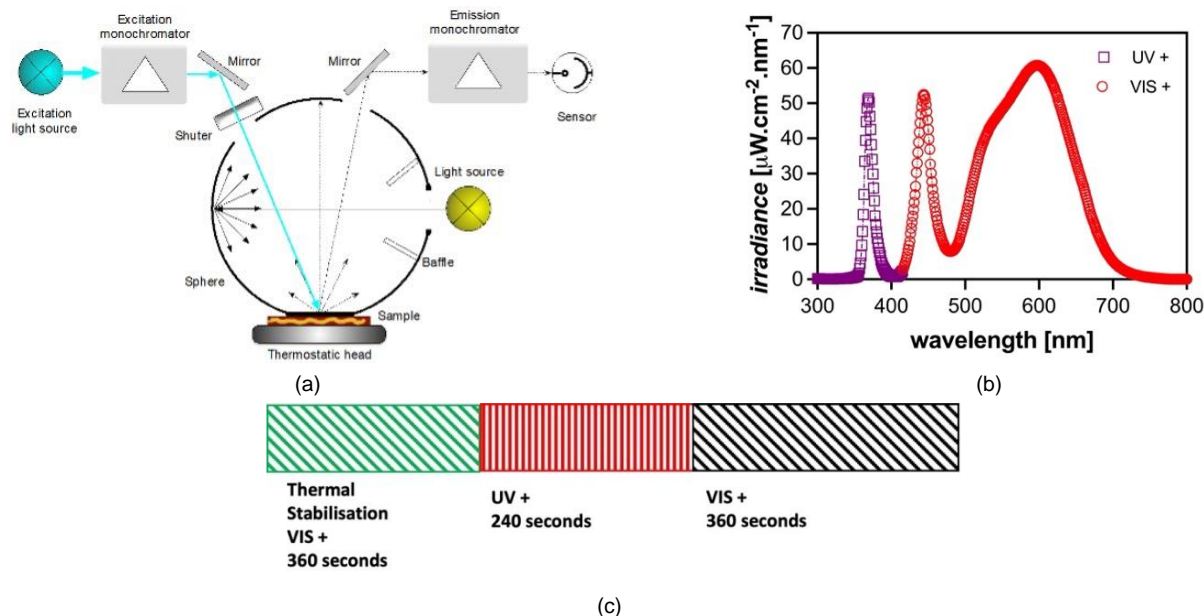


Figure 3. Schematic illustration of a) FOTOCHROM3 spectrophotometer, b) SPD curves of used light sources, c) Fatigue scenario used under continual UV irradiance.

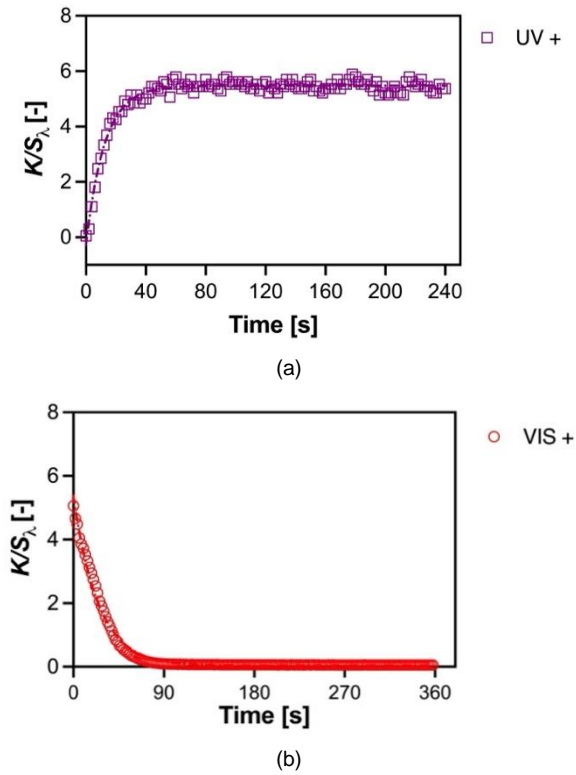


Figure 4. Photochromic cycle: a) Growth phase with one-phase association fit, b) Decay phase with one-phase dissociation fit.

$$K/S_{\lambda} = \frac{(1-R_{\lambda})^2}{2R_{\lambda}}, \quad (1)$$

where K/S_{λ} – where K_{λ} is the absorption coefficient, and S_{λ} is the scattering coefficient of the photochromic dye observed at its dominant wavelength in the visible spectra of an electromagnetic spectrum

The photo-kinetic behaviour of the photochromic system is analysed using the first-order kinetic model, as demonstrated in earlier research that investigated the kinetic behaviour of several photochromic dyes and pigments [8-13]. This behaviour is described by Equation 2.

$$K/S_{(t)} = \left(K/S_{(0)} - K/S_{(\infty)} \right) \exp(-kt) + K/S_{(\infty)}, \quad (2)$$

where, the subscript t - actual time, 0 - at the beginning of the reaction (photo colouration phase), ∞ - time at infinity (reaching photo-steady equilibrium state, upper plateau and lower plateau for the growth phase and decay phase, respectively), k – rate constant [s^{-1}], t – UV irradiance time used in [s] during the growth phase for each photochromic cycles (n), $K/S_{(\infty)}$ – the upper plateau obtained after fitting the raw data of each growth phase cycles, (n) – is the growth phases of each photochromic cycle.

Equation 2 represents a mathematical model that examines the relationship between photochromic systems' growth and decay phases, thus following the first-order kinetics model. It is used to study the photo-fading behaviour of these systems under

observed cycles of UV irradiation. Nevertheless, this study specifically examines the photo-fading characteristics of development phases, which are assessed across multiple cycles of UV irradiation utilising various modes. The photochromic system exhibits a photo-fading behaviour when subjected to several UV irradiation cycles during consecutive measured growth stages. Thus, the quantity of UV irradiance cycles is denoted as the converted UV dosage required for photo-fading photochromic dye molecules. The data processing begins by converting the acquired reflectance data into colour intensity values at their most prominent wavelength using the Kubelka-Munk (K/S) function, as described in Equation 1. Subsequently, the data is further processed for evaluation using Equation 2, as previously mentioned.

The photochromic system's dominant wavelength $\lambda_{max} = 570$ nm experiences a transition in colour, shifting from a colourless state to a vivid, deep purple hue. The colour span values were adjusted to a one-phase association model based on its dominant wavelength to calculate the kinetic parameters: rate constant k [s^{-1}], half-life ($t_{1/2}$) and maximum colour span value (K/S_{max}) for the photocolouration phase. Similarly, a one-phase dissociation model was used to analyse the decolouration phase. Observations have been made on the cycles of recorded UV irradiance, the spans acquired, and the behaviour of the plateau in the extended continuous mode. The photodegradation behaviour adheres to a one-phase dissociation model for all measured cycles. Furthermore, we note that the photodegradation behaviour is consistent for the UV mode without a relaxation phase, in which the photochromic system is solely exposed to UV light. The raw data was processed and analysed using the GraphPad 10 (10.3.0) statistical software on the MacOS platform.

RESULTS AND DISCUSSION

The study examines the spectrokinetic analysis of a photochromic dye based on spironaphthoxazine. The photochromic system was created as photochromic prints utilising the screen-printing technique. The photochromic dye molecules are deposited on the surface of the textile fabric. We obtained the kinetic measurements using a specialised spectrophotometer known as FOTOCHROM3. This instrument allows us to observe the photoreaction of a photochromic system as it undergoes the growing phase (photocolouration) and decay phase (decolouration) of a general profile curve in a photochromic cycle. We performed the photoexcitation using a monochromatic light source, specifically ultraviolet (UV) light, with a wavelength of 360 [nm]. We conducted the thermal relaxation of a photochromic device by exposing it to visible light from a continuous irradiance source. In the UV irradiance mode, the fabric absorbs the intensity of

the UV light, which partially penetrates and bends back due to its structure. The FOTOCHROM3 device measured the reflectance values of each photochromic system, specifically capturing the changes in photocoloration and decoloration. This measurement was done under the continuous irradiance mode, with the growth and decay phases seen over a specific period.

The reflectance vs. time curves from the FOTOCHROM3 spectrophotometer were used to process the data for each photochromic system using the stepwise method described in the data treatment section. Figure 5 presents the K/S value of each photochromic system at its dominant wavelength during the growth and decay phases, as determined by fitting the one-phase kinetic model. We determined the kinetic parameters by fitting the spectrum data using the one-phase association and dissociation model to analyse the growth and decay phases.

The rate constants for photocoloration and decoloration of each photochromic system are graphed in Figure 6. Furthermore, the half-life values of each photochromic system throughout its growth and decay phases are graphically represented in Figure 7. The colour span values of each photochromic system and their dependence on the dye concentration are linear, as illustrated in Figure 8. The measured energy of the UV irradiance area is 7.9 W.m^{-2} , whereas for the visible region, it is 97.79 W.m^{-2} . The diameter of the aperture for irradiance during excitation and relaxation is 28 mm , and the exposure area is 0.0006154 m^2 . The exposure phase lasted 240 s , and the decay phase lasted 360 s . The observed photon flux for UV+ and VIS+ is defined as $\Phi_{\text{UV+}}$ and $\Phi_{\text{VIS+}}$, and the estimated values for $\Phi_{\text{UV+}}$ are $1.953 \times 10^{22} \text{ photons.m}^{-2}$ and $\Phi_{\text{VIS+}}$ are $2.390 \times 10^{23} \text{ photons.m}^{-2}$, respectively.

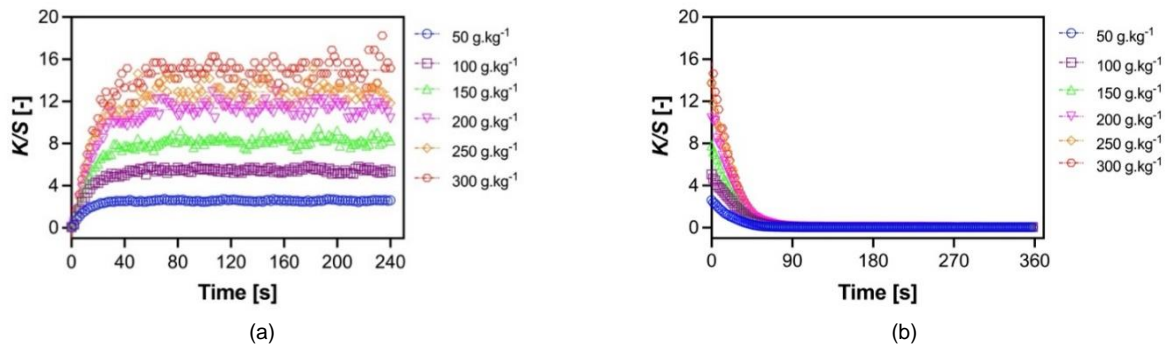


Figure 5. The photochromic system's: a) Growth phase with one-phase association fit, b) Decay phase with one-phase dissociation fit.

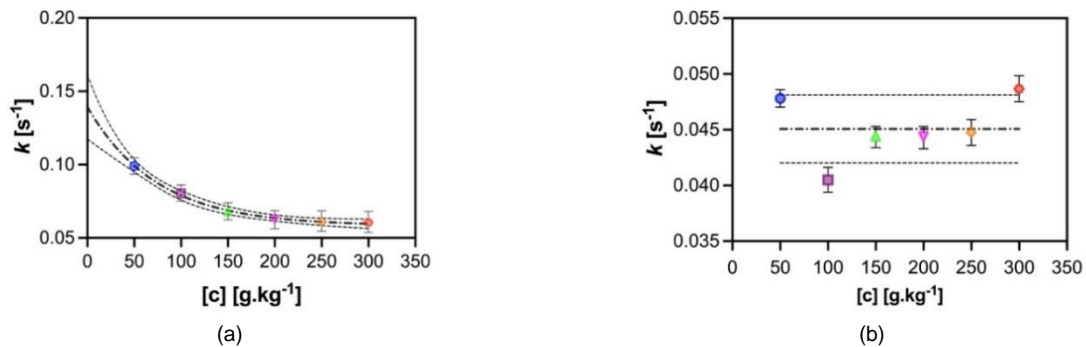


Figure 6. The photochromic system's: a) Growth phase rate constant, b) Decay phase rate constant under continual irradiance mode.

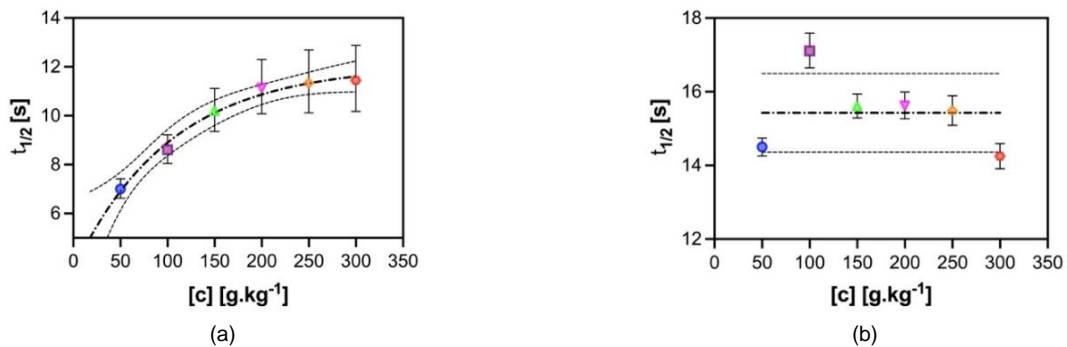


Figure 7. The photochromic system's: a) Growth phase half-life (s), b) Decay phase half-life under continuous irradiance mode.

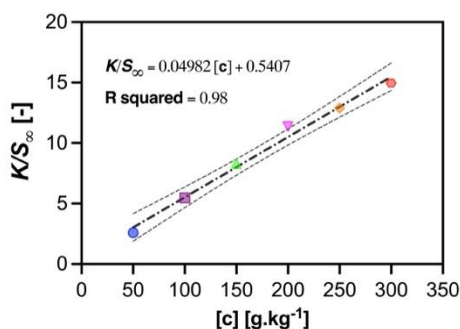


Figure 8. Dependency of photo colouration value vs. the concentration of photochromic dye under continuous UV irradiance.

CONCLUSION

The research study provided relevant information regarding photoreaction kinetics in a photochromic system. This reaction takes place under continuous irradiance mode and involves the process of photo colouration and decolouration. The reflectance vs. time curves produced during the photochromic cycle were used to analyse this process. The photochromic dye has a half-life of around six seconds when it is in a state of photo-excitation and a half-life of two seconds when the dye molecules are decolourising. The spiroxazine dye in textile print form was exposed to a continuous and uniform beam of monochromatic light. The light source used had a 360 nm wavelength and was stimulated by UV radiation. The measurements were taken at a temperature of 293.15 ± 2 K. Nevertheless, the transformation from a colourless state to a coloured one in the photoreaction is contingent upon the wavelength of light, namely within the visible spectrum, where the majority wavelength is found. Photodecolouration also occurs at the most prominent wavelength in the visible spectrum. The colour span values represent the highest colour intensity achieved during the phase of colour change caused by light exposure. These values are derived by analysing each photochromic system's reflectance versus time curves. The photodegradation during a single kinetic measurement is insignificant, as it only occurs and can be detected during a series of UV irradiation cycles during the growth phase of a photochromic cycle. The rate constants for photocoloration and decolouration adhere to the decay model, with the former increasing as the dye concentration within the photochromic system increases, while the latter remains relatively constant. The colour yield of the photochromic system is directly proportional to the dye concentration within the system, resulting in a linear relationship.

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