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Yellowing of UV varnishes with focus on its temporal behaviour and correlations between intensities and degrees of polymerisation

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Abstract

This work discusses the curing-induced discolouration of UV varnishes, the so-called ‘yellowing’. A study of 47 market available acrylic-based UV varnishes from five manufacturers shows that all varnishes exhibit significant yellowing. In order to clarify if yellowing can be exploited for monitoring the cure of the varnishes, three varnishes are analysed in detail, including one low-migration and one photoinitiator-free self-initiating formulation. For this purpose, the applied varnishes are measured with respect to yellowing as well as their actual polymerisation degrees. Both measures are checked for correlations to develop a novel method for monitoring the degree of polymerisation based on colourimetric measurements. Additionally, the temporal behaviour of the yellowing is determined as it is of significant importance when considering a practical usage of the yellowing approach.

Keywords: UV curing, temporal yellowing, discolouration, colourimetry, FTIR-ATR spectroscopy

1. Introduction and background

1.1 Background

A relevant number of print products are UV-varnished to achieve various optical and barrier properties. In some applications, like narrow web label printing, UV curing has reached a predominant position (Etiketten Labels, 2020). Thereby, the polymerisation degree (colloquially spoken: UV curing degree) is of fundamental importance in terms of quality and safety.

From a quality perspective, a certain polymerisation degree is necessary to ensure a good product quality, especially if it comes down to post-press processing aspects. An insufficient curing degree, for example, could cause blocking in the delivery stack or the reel. When considering product safety, within an insufficiently polymerised varnish layer, especially unaffected photoinitiators might tend to migration and could subsequently lead to health/safety issues for operators or even consumers (Green, 2010). Even though the latest-

findings indicate that no hazard for consumers is to be expected from sheetfed offset printed products, process control and an adequate degree of polymerisation is still in the interest of all parties involved (bvdm, 2020). For these reasons, several methods to check the quantity/quality of the polymerisation exist in industrial practice. Among them are some methods based on manual tests as well as device-supported methods (ref. section 1.3). Bohn, et al. (2017) presented an alternative approach for indirect cure monitoring based on spectral measurements of the curing-induced discolouration of UV varnishes, the so-called ‘yellowing’. They found a correlation between yellowing intensity and exposed UV dose for three common photoinitiators solved in acetone. Furthermore, a correlation between yellowing and actual polymerisation degrees for two laboratory UV varnishes could be shown.

As a logical extension of these findings, this research focuses on industrial varnish systems to determine whether findings of Bohn, et al. (2017) are relevant for industrial print processes. By investigating 47 varnish

systems available on the market, it is the aim to clarify if the yellowing is a relevant variable in commercial varnishes used. Next, three varnishes are analysed in detail to verify a significant correlation between yellowing and the actual polymerisation degree. Also, the temporal progression of the yellowing is examined colourimetrically as a change in yellowing intensities over time. This aspect becomes relevant when yellowing is measured as a process parameter. The studies described are fundamental on the way towards a practical realisation of the yellowing approach as a novel method for monitoring UV curing processes by using common spectrophotometers in the visible spectrum.

1.2 State of the scientific knowledge

The technical principles of UV curing, its related inks and varnishes and other relevant process parameters are widely described in literature, for example in Glöckner, et al. (2008). Therefore, only a brief abstract of UV curing principles and the background of yellowing in particular is given in the following.

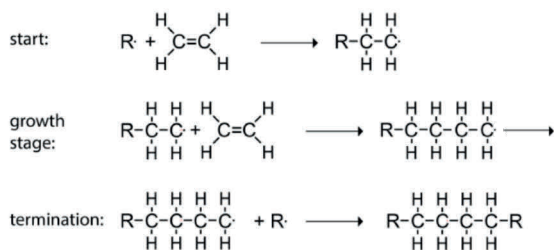


Figure 1: Schematic diagram of the radical polymerisation process (Bohn, et al., 2017)

Most UV varnishes relevant to printing industry are based on acrylates and mainly consist of reactive diluents, binders and photoinitiators. During the process of radical polymerisation, photoinitiators cleave under the influence of UV radiation and form free radicals R· in the start phase. These so-called ‘start radicals’ initiate the actual polymerisation process by breaking the C=C double bonds of the acrylic monomers and oligomers, which then crosslink in the growth stage to form polymers (Glöckner, et al., 2008). This process ends when all free radicals have reacted. Figure 1 shows the polymerisation process schematically. Besides the free radicals, the UV-induced cleavage of a photoinitiator can result in different photoproducts due to secondary reactions, further scissions and combinations of radicals with parts of the formulation (Green, 2010). These photoproducts have no supporting influence on the polymerisation but can cause a visible discolouration by their specific absorption characteristic in the UV- and the adjacent visible blue region of the electromagnetic spectrum. The findings of Bohn, et al. (2017) are in accordance with increasing yellowing intensi-

ties for increasing UV doses observed by Studer and Königer (2001), examining a photoinitiator in combination with a binder. However, this connection is only valid for lower UV doses (less than ~2000 mJ/cm²) as the photoproducts, for their part, are sensitive to UV light (Studer and Königer, 2001). Schwalm (2006) also points out that the yellowing can decrease at higher doses. Furthermore, the quantity of formed photoproducts very much depends on the particular photoinitiators and is also proportional to the photoinitiator(s) concentration, relative spectral power distribution of the used light source relative to the photoinitiators absorption spectra, and film thickness (Green, 2010). Schwalm and Green exemplarily showed, that reaction processes can lead to the formation of a broad variety of photoproducts for different photoinitiators. In addition, all other varnish components and even the used substrate as an (unwanted) supply of additional reaction products, might have an impact on the yellowing intensity (Schwalm, 2006; Green, 2010). Due to this potential multicausality, yellowing of a formulation is hardly predictable for a specific application. In this work, the term ‘yellowing’ is always referring to the UV-curing-induced initial photoyellowing, which needs to be distinct from the yellowing of polymers due to long term aging. The initial yellowing is described as a decrease of certain absorption bands within the first hours after curing (Schwalm, 2006). Figure 2 exemplarily shows the spectral reflectance of a UV varnish on a white substrate in its unpolymerised as well as its polymerised state for two UV doses. The polypropylene (PP) discolouration visible here can be attributed to the varnish, as the substrate does not show significant UV-induced discolouration (cf. section 2.2).

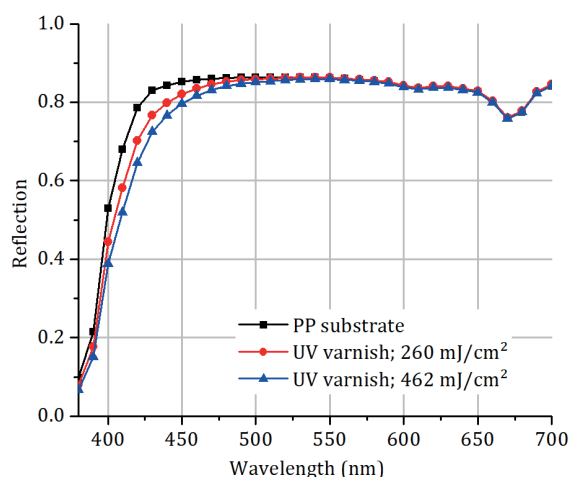


Figure 2: Spectral reflectance of an UV varnish applied on a white PP substrate polymerised with two UV doses; the polymerisation induced yellowing can be seen between 400 nm and 530 nm as a reduction in the reflectivity, i.e., an increased absorption

1.3 Methods to check the polymerisation quantity/quality

Various existing industry-relevant manual methods, such as solvent rub test, tape test, thumb twist test, powder test, and potassium permanganate test, to check cured layers are widely used and described in literature (Glöckner, et al., 2008). Except the last one, all of these have in common that they check different surface properties of a cured layer which are not necessarily related to the actual polymerisation degree. These methods, from some points of view, can be seen as inadequate gap fillers in the absence of a direct measurement of the degree of polymerisation. Besides manual techniques, some device-based methods are available to check various properties of cured layers. Hubergroup offers the NewV cure UV VIS spectrophotometer including the related method and special varnishes. The varnishes contain a marker substance, which is extracted after curing into a liquid. The quantity extracted is determined using the spectrophotometer and represents the curing degree (Schröder, 2019). Fogra offers an automated acetone test called ACET and the Fogra Ushio Cure Check which uses heat and pressure to check cure quality (Rauh, 2007). The UV CURE CHECK of PITSID (2019) determines the coefficient of sliding friction as an indicator for surface cure.

1.4 FTIR as a direct method to determine polymerisation degrees

For molecular spectroscopy purposes like classification and quantification, the Fourier-transform infrared (FTIR) spectroscopy as an analysis technique is widely used. Based on the molecule's interaction with infrared radiation, FTIR allows identification of material compositions, concentrations and functional groups, for instance. Using FTIR in attenuated total reflection (ATR) mode, one can conduct reflection measurements of both solid and liquid samples. The FTIR spectroscopy is a standard method to determine polymerisation degrees on a laboratory level. During the polymerisation process, the C=C double bonds of acrylic molecules split and the monomeric and oligomeric molecules crosslink to polymers. Consequently, one can determine the actual polymerisation degree of a UV varnish layer as a function of the remained C=C double bonds compared to an uncured varnish. The C=C double bonds have distinct absorptions at $\sim 810\text{ cm}^{-1}$ whose intensity is proportional to the number of double bonds contained in a varnish (Glöckner, et al., 2008). To compensate variations in the measurements, it is necessary to perform an internal calibration for each measurement using a spectral band which is not altered by curing. In case of acrylates, the C=O bond absorption at $\sim 1720\text{ cm}^{-1}$ fulfils this requirement (Fernández-Francos, et al., 2013). For quantification of a species, the

peak heights in the absorbance spectra are determined, starting from their specific baselines. Out of these peak heights, the actual degree of conversion x is calculated by Equation [1] (Fernández-Francos, et al., 2013).

$$x = 1 - \frac{(A_{\text{spec}}/A_{\text{ref}})_{\text{cured}}}{(A_{\text{spec}}/A_{\text{ref}})_{\text{uncured}}} \times 100 \quad [1]$$

where x is a degree of conversion in %, A is absorbance, 'spec' denotes sample species (C=C bond at $\sim 810\text{ cm}^{-1}$), 'ref' denotes sample reference (C=O bond at $\sim 1720\text{ cm}^{-1}$), 'cured' and 'uncured' denotes UV irradiated sample and not irradiated sample, respectively.

Figure 3 shows an FTIR-ATR absorbance spectrum of a commercial UV varnish in uncured and cured state. The relevant wavelength bands for quantification of the conversion are marked.

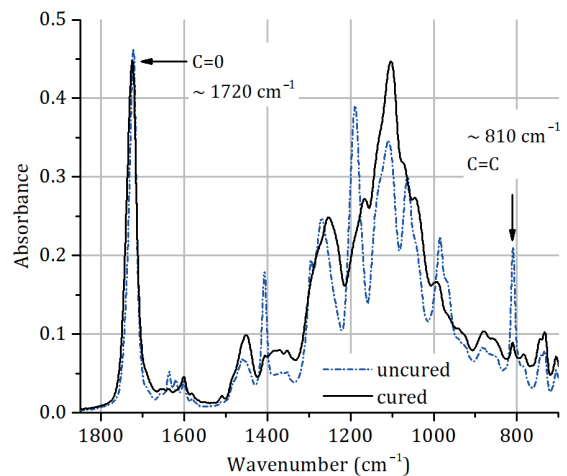


Figure 3: FTIR-ATR absorbance spectra of a UV varnish in uncured and cured state, the crucial bands at 1720 cm^{-1} and 810 cm^{-1} to quantify polymerisation are marked

For practical users of the UV curing, e.g. printers, this method is often seen as an elaborate, cost-intensive and laboratory-only method. For these reasons, it is not directly used in print production, but utilised by some larger companies on a quality assurance level. Thanks to appropriate software, it is just as easy to determine polymerisation with an FTIR as it is to determine colourimetric quantities with a spectrophotometer. Therefore, in the opinion of the authors of this article, at least the complexity aspect is worth a discussion.

2. Methods

2.1 Materials

In total, 47 samples of typically distributed UV varnishes have been obtained from five European varnish producers for conventional curing with medium

pressure mercury lamps and application via flexo print units. Our goal was to obtain a broad field of samples, representing current practice. The samples include high glossy and matte varnishes and all intermediate levels, varnishes for special application like anti-static protective varnishes and finishing of digital prints, varnishes for thermal transfer, embossing and several other demands. Low-migration (LM) systems are among these varnishes and also systems which are free of additional photoinitiators but containing self-initiating monomers. We assign a number to each varnish and provide Table 1 with basic characteristics like LM, glossy and matte. As a concession to the supplier, we do not provide brand names. Out of this range, three varnishes are selected for a deeper investigation of actual polymerisation degrees as well as the temporal progression of their yellowing (highlighted in Table 1). These varnishes were selected in order to represent different categories and yellowing intensities from our sample field.

Table 1: Sample field of 47 UV varnishes with allocated ID, manufacturer (Manuf.) and basic characteristics (Charac.) glossy (g) and matte (m), LM and self-initiating LM (LM+)

ID	Manuf.	Charac.	ID	Manuf.	Charac.
1	A	g	25	B	g
2	A	g	26	B	m
3	A	g	27	C	g
4	A	g	28	C	g
5	A	g, LM	29	C	g, LM
6	A	g	30	C	m, LM
7	A	m	31	C	m
8	A	m	32	D	g, LM
9	A	g	33	D	g
10	A	m	34	D	g, LM
11	A	g	35	D	g
12	A	g	36	D	g
13	A	m	37	D	g
14	A	m, LM	38	D	g
15	A	g, LM	39	D	m
16	A	g, LM+	40	D	m
17	A	g, LM+	41	E	g
18	A	m, LM+	42	E	g, LM
19	A	g, LM+	43	E	g, LM
20	A	g, LM+	44	E	g
21	B	g, LM	45	E	g
22	B	g	46	E	g
23	B	g	47	E	m
24	B	g	/	/	/

2.2 Application

For the wide investigation of the 47 varnishes, application is done with a 12 μm rod wire in order to enable an efficient sample generation. For the investigation

of correlations and temporal progression, an IGT Printability Tester F1 in flexo mode is used. Its anilox roller has a nominal pick-up volume of 16 ml/cm². All varnishes are applied on a white 250 μm PP substrate, which is not affected by UV radiation in its colour (white point for M2 mode, D50, 2°: CIELAB 94,1; -2,04; 0,94). The tests were carried out under laboratory conditions. All samples have been stored at 21 °C under exclusion of light between measurements.

2.3 Colourimetric analysis

The colour measurements are done with a Konica Minolta FD-7 spectrophotometer in 45°/0° geometry, M2 measurement mode, D50 illuminant, 2° CIE standard observer, 10 nm wavelength pitch, 3 mm measuring spot configuration. According to its spectral characteristic, yellowing can be expressed within the CIELAB colour space as change of the b^* coordinate. Calculation of Δb^* occurs in accordance with ISO/CIE 11664-6:2014 (International Organization for Standardization, 2014). For measuring the temporal progression of the yellowing, the time gap between curing and first measurement is fixed to ten seconds.

2.4 FTIR-ATR readings

A PerkinElmer Spectrum Two FTIR with ATR accessory is used to determine the acrylate conversion. The spectrometer measures in a spectral bandwidth of 8300–350 cm⁻¹ (1204–28571 nm) and uses a versatile diamond crystal in the ATR top plate. The macros for an automated quantification process are written with PerkinElmer Spectrum Quant software. Each sample is measured three times whereby each measurement automatically consists of the average of four single measurements. Each sample is measured for yellowing and polymerisation at identical spots.

2.5 UV conveyor belt dryer

For curing the samples, the laboratory UV unit Technigraf AKTIPRINT MINI UV equipped with a standard medium-pressure mercury UV lamp is used. The emission spectrum of this type of lamps can be seen in relevant literature, for example in Green (2010). The UV source emits an irradiance of 441 mW/cm² in the entire UV range for an electric input power of 120 W/cm. We use UV doses from 462 mJ/cm² to 110 mJ/cm².

Variations of the UV doses are realised by different conveyor belt speeds in a range of 20 m/min to 4 m/min. Both irradiance and the resulting UV doses are measured with an Opsytec UVPAD E spectral radiometer. This device covers a wavelength range from 240 nm to 480 nm. We only consider the UVC, UVB and UVA range up to 400 nm for power data.

3. Results

3.1 Yellowing of market available UV varnishes

Figure 4 shows all analysed varnishes in their unpolymersed and their polymersed state for two UV doses. Inherent colours of all varnishes differ from the substrate’s white point and all varnishes show significant yellowing with clearly different intensities within the sample field. For further evaluation we define Δb_i^* as the inherent varnish colour and Δb_c^* as the yellowing intensity for a UV dose of 462 mJ/cm², related to Δb_i^* .

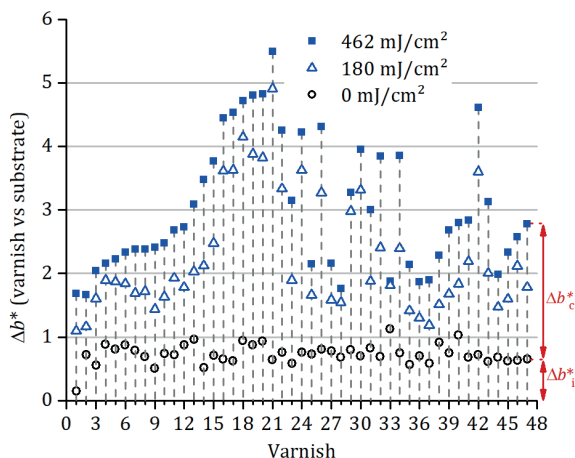
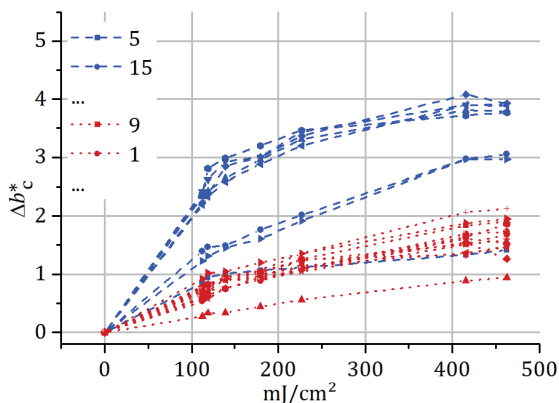


Figure 4: CIELAB Δb^* values of 47 UV varnishes for three UV doses of 0, 180 and 462 mJ/cm² (141 samples), Δb_c^* and Δb_i^* are exemplarily indicated for varnish 47

Our knowledge of the varnishes consists only of the manufacturer’s characterisation and is therefore limited to the properties we described in the materials section. During the analysis, we find the greatest yellowing intensities in the sample field for LM varnishes. Figure 5 shows diagrams containing all varnishes from two manufacturers, including 10 LM and 15 non-LM



varnishes. The LM systems are shown with blue dashed line, non-LM with red dotted lines. These findings are representative for the other 22 varnishes of three manufacturers. We have reduced the legend of the left diagram and show results in two diagrams for better clarity.

The sample field can also be categorised by the surface properties glossy or matte. Figure 6 shows in two diagrams the varnishes already depicted in Figure 5, but here we differentiate between glossy (blue dashed lines) and matte (red dotted lines) varnishes. Based on this categorisation, no pattern can be identified. We could not identify any other categorisation.

In Table 2, the sample field is categorised by LM, non-LM, glossy and matte varnishes and the yellowing is quantified by mean Δb^* values in order to highlight our findings. The glossy varnishes surpass the matte ones in their average yellowing intensity. This is due to the fact that most of the LM varnishes are categorised as glossy and, thus, significantly raise the mean value. To compensate for this distorted representation, we have added the categories glossy and matte excluding LM varnishes.

Table 2: Yellowing properties of the sample field based on arithmetic mean of Δb_i^* and Δb_c^* values, categorised by LM and surface properties glossy and matte

Varnish category (quantity)	$\bar{x} \Delta b_i^*$ (substrate vs varnish unpolymerised)	$\bar{x} \Delta b_c^*$ (varnish unpolymerised vs polymerised)
all (47)	0.73	2.29
LM (15)	0.73	3.33
non-LM (32)	0.73	1.80
glossy (35)	0.73	2.37
matte (12)	0.74	2.05
glossy, non-LM (23)	0.72	1.89
matte, non-LM (9)	0.77	1.89

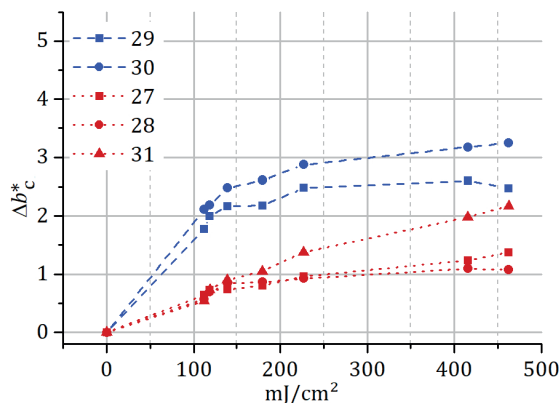


Figure 5: CIELAB Δb_c^* values of 25 UV varnishes, non-LM (dotted) and LM (dashed), from two manufacturers for eight UV doses ranging from 0 to 462 mJ/cm²; LM varnishes show the trend for higher yellowing intensities

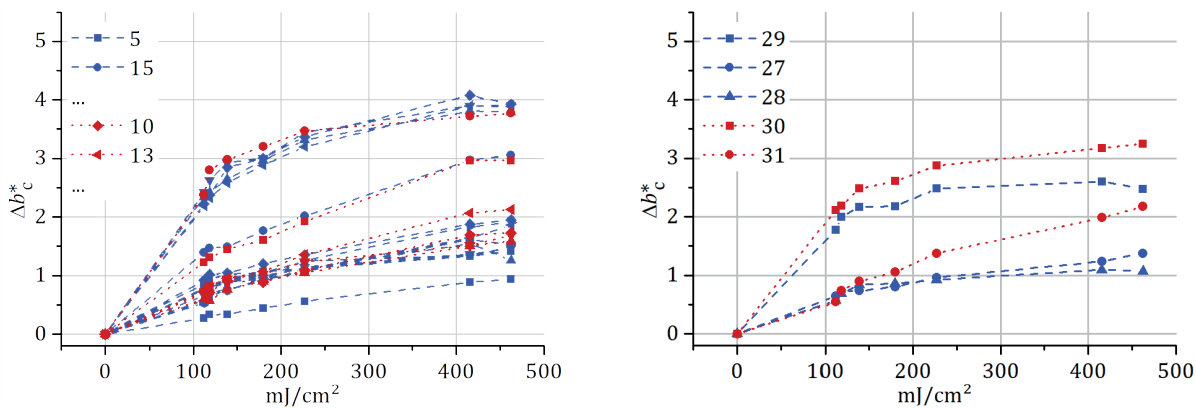


Figure 6: CIELAB Δb_c^* values of 25 UV varnishes, matte (dotted) and glossy (dashed), from two manufacturers for eight UV doses ranging from 0 to 462 mJ/cm^2 ; no trend can be identified

Figure 6 shows that for some varnishes even an increase of the UV dose from 415 mJ/cm^2 to 462 mJ/cm^2 does not lead to an intensified yellowing. The same can be observed for varnish 5 and 19 in Figure 7 (cf. section 3.2), where the three crowded data points at the upper end of the regression line indicate that increasing the dose does not further increase polymerisation. This is because the varnishes have reached their polymerisation maximum for our curing condition.

3.2 Correlation between yellowing and polymerisation

Figure 7 shows the relationship between b^* value and polymerisation degree for three varnishes cured with 15 different UV doses. The varnishes differ significantly in yellowing intensities, but linear correlations are apparent for all of them. Based on Figure 7, one can ascertain a significant relationship between the acrylate’s conversion and discolouration of the UV varnishes.

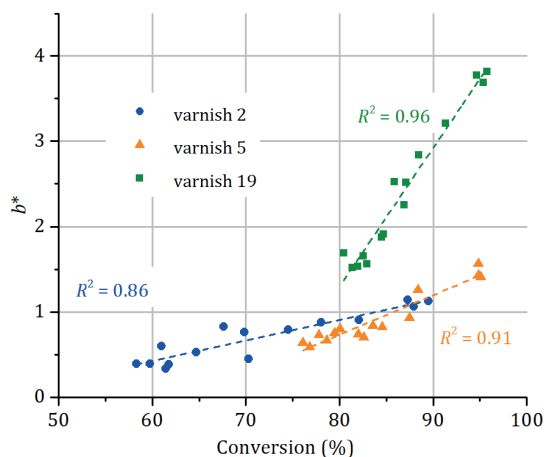


Figure 7: Correlation between yellowing as CIELAB b^* value and acrylates conversion for three UV varnishes cured in 15 UV doses ranging from 110 to 462 mJ/cm^2 ; coefficients of determination R^2 are given

Despite high coefficients of determination R^2 values, deviations from the ideal linear course can be seen. The sample generation and the measurements have multiple potential sources of errors like variations in varnish layer thickness, measurement of yellowing and polymerisation at non-identical spots, white point variations of the substrate, influence of morphology changes on colour measurements, penetration depth of the ATR measurements and measuring device errors. We do not conduct a complete error analysis as we primarily want to determine whether there is a fundamental connection between yellowing and polymerisation or not. Regardless of any measurement errors, a linear correlation can be established between the two variables. Of course, measurement errors are crucial in questions of practical measurability. We are aware of potential sources of errors and will consider them in a future validation of the approach.

3.3 Temporal behaviour of the yellowing

Figure 8 shows the temporal progression of the yellowing of varnishes 2, 5 and 19 for a period of 24 hours after initial curing.

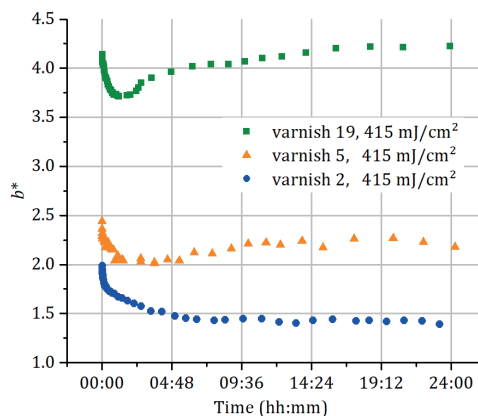


Figure 8: CIELAB b^* values for UV varnishes 2, 5, 19 for a time range of 10 s to approx. 24 h after curing

In the first two hours after curing, the measurement intervals are small as the yellowing changes rapidly, while intervals become greater with progressing time. When comparing the trends of the varnishes, clear differences can be seen. While varnish 2 shows an exponential decay, varnish 5 decreases in the first two hours and then, after two hours of stagnation, increases again to approach its initial b^* value. Varnish 19 shows this effect intensified and even reaches its initial yellowing after about 19 hours. All varnishes remain stable after a period of 24 hours.

4. Discussion

In the following, the results are discussed successively, each followed by an estimation of its relevance to the yellowing approach.

Although yellowing is undesirable and manufacturers have been searching for non-yellowing formulations for decades, all used varnishes show a curing-induced distinct yellowing with a mean Δb_c^* of 2.29. One can also see significant Δb_i^* values due to the application of the unpolymerised varnishes to the substrate. As well as laminations and other coatings, the presence of a varnish layer influences the visual and measurable colour due to several optical effects like scattering, refraction and absorption. However, from a colorimetric point of view these effects mainly influence the brightness and not the chromaticity (Stiene, Urban and Rodriguez-Giles, 2019). Therefore, the changes in b^* values can be attributed to the inherent colour of the varnishes.

With regard to yellowing, especially the 15 LM systems stand out by showing the highest intensities with a mean Δb_c^* of 3.33, compared with a mean Δb_c^* of 1.80 for non-LM varnishes. In general, the reasons for the yellowing potential of a varnish are to be found in the chemistry of its components and are not subject of this work. However, it seems conceivable that it is more difficult for manufacturers to focus on low-yellowing LM-formulations because there are less permitted photoinitiators for LM systems. The chemical industry will certainly continue research here.

The intensive yellowing signal of modern LM systems contributes to the yellowing approach in a way that a good measurability is crucial for the approach. As product safety is always an issue to be improved, LM varnishes are expected to gain market share. In addition, until there is certainty about the hazard potential of UV varnishes, the use of LM systems is also in line with the precautionary principle. Analysing the category glossy/matte, no pattern can be identified. In the absence of more detailed information about the composition of the varnishes we could not identify other

categories. However, a precise knowledge of the composition presumably would allow us to draw closer conclusions about the interaction of different components regarding to their yellowing.

Based on our findings, we can state out that curing-induced yellowing is a potentially relevant parameter while working with UV varnishes, in particular against the backdrop of rising demands on colour quality.

Linear correlations between yellowing and polymerisation can be seen in Figure 7. As already mentioned at the end of section 3.1, in Figures 5, 6 and 7 we can see maximum yellowing and polymerisation, which we cannot increase further with our curing conditions. In other conditions, e.g. in an inert environment or using higher irradiances, the particular polymerisation maximum could potentially be increased. Thinking about a novel approach for monitoring UV print productions based on colour measurements, a reliable measurability is crucial. The weak yellowing of varnish 2 could make it difficult to realise measurements for this particular varnish. This must be investigated in further research under practical conditions taking into account an error analysis.

The temporal yellowing progressions of the three different varnish systems do not allow general statements for conventional or LM systems in their entirety. However, they demonstrate exemplarily that yellowing is a dynamic effect in the first 24 hours after curing and can go well beyond a simple decay after cross-linking. Especially in the time right after curing, the yellowing alters quickly. Since the time gap between curing and measurement is ten seconds, it can be concluded that the yellowing is even greater directly in the delivery of the dryer. This short period of time is probably not relevant for most practical processes, but may have an influence when working with inline colour measuring devices and comparing to manual measurements at the printing press. We have already described above that a varnish as an additional layer has an influence on the colour appearance. Furthermore, and that's characteristic for UV varnishes, yellowing can bring in a specific discolouration, which is not limited to changes in brightness and also is time-dependent. Hence, concerning colour measurements of UV cured products, the intensity and temporal behaviour for a specific varnish as well as the time of measurement should be considered to obtain consistent colour measurements.

5. Conclusions

This investigation shows significant yellowing for all varnishes considered. The yellowing is a dynamic effect that shows changes in the first 24 hours after the initial

curing. For one varnish it decreases within a few tens of minutes in a nearly exponential manner and then remains stable, whereas other varnishes show a yellowing rebound a few hours after an initial decrease. Linear correlations between yellowing intensity and degree of polymerisation are exemplarily determined for three UV varnish systems of the categories conventional, low-migration and self-initiating low-migration. There are no indications why linear correlations should not be valid for other common varnishes. The yellowing intensity of a varnish also depends on its layer thickness. Following Lambert-Beer law, this connection is of logarithmic

character. Knowledge of the influence of both polymerisation and layer thickness on yellowing intensities opens the possibility to predict yellowing for any layer thickness and degree of polymerisation. To achieve this objective, future work could investigate the measurability of yellowing in practical processes, take a closer look at correlations under different circumstances, and, fundamentally, deal with a possible implementation of the yellowing approach. Another interesting field is the discolouration of UV inks. Considering what is known so far, beneficial use of the actually unwanted yellowing of UV varnishes seems possible.

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