Artificial Ageing of Wood Finishes Monitored by IR Analysis and Color Measurements

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ABSTRACT: Photocurable finishes appear now as very attractive products for industrial wood joinery for reducing volatile organic compounds (VOC). Nevertheless their poor durability is currently reported. In this study, we aimed to enhance stability of advanced formulations based on photoinitiators working under UV-visible light by introducing UV absorbers, the absorption band of which is shifted compared to the band of initiators. We tested the efficiency of these stabilizers for two curable coatings applied onto oak and spruce. Modifications of samples at molecular and macroscopic scales were quantified after an accelerated ageing process by FTIR spectroscopy, measurements of color variations, apparition of cracks. It appears that both UV absorbers tested do not greatly improve the durability of systems and the results obtained allow to better understand the causes of the poor characteristics of the finishing systems. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1938–1946, 2012

Key words: wood coatings; photopolymerization; UV initiators and absorbers; artificial ageing; FTIR microscopy; color measurements

INTRODUCTION

Wood is a complex material which can be quite easily photo-degraded because of light absorption by chromophoric groups supported on lignin.[1,2] Among the solutions that can be proposed to protect wood against photodegradation,[3,4] the easiest is to apply finishes such as paints, coatings, varnishes etc. Photocurable finishes[5,6] are now more and more used in industrial wood joinery and their use should further develop in the coming years. They are composed basically of photoinitiators and a mixture of monomers and/or oligomers dissolved into reactive diluents (i.e., UV curable multifunctional monomers).[7] The latter are then directly involved in the curing process and are "trapped" in the cured film. Compared with classical polluting solvent-borne systems, waterborne or powder photocurable formulations are one solution to comply with restrictions imposed by legislation to reduce the emission of volatile organic compounds VOC, from coatings.[8,9] Nevertheless some technical problems are reported while using UV photocurable finishes. Indeed, the photochemical reactivity of some wood components

with radical species retards and inhibits the initiation phase of the curing process[10,11] and also has an influence on the degree of cure and the mechanical properties of the finish film.[12] Furthermore, the addition of UV absorbers into such photocurable systems is difficult because these compounds may interact with photoinitiators and retard the polymerization process since the spectral windows of both types of substances are largely superimposed. To overcome this difficulty, some researchers proposed to implement photoinitiators working under visible light.[13] UV absorbers are generally organic or inorganic substances such as oxanalide, benzophenone, benzotriazole, triazine, nanodispersed titanium, or cerium dioxide which preferentially absorb and/or diffuse UV radiation arriving onto the sample surface.[14–16] They are often incorporated—alone or combined—into finishes to enhance the durability of the cured coating.[17] Thus solutions have to be found to increase the durability of UV curable finishes, particularly for outdoor joinery which is submitted to severe climatic conditions. In our previous work[18] we sought to develop clear photocurable formulations containing specific photoinitiators able to initiate the curing process under visible light so that UV absorbers could be incorporated into the finish. The curing process was studied by means of photocalorimetry and it was observed that the addition of UV absorbers did not inhibit the initiation phase when photopolymerization occurred at wavelengths above 400 nm.[19] In this study we investigate the protective effects of UV absorbers by following the

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TABLE I
Ageing Conditions Applied for Testing Samples

Step		Temperature	Duration	
1	Condensation	45°C	24 h	
2	Subcycle steps 3+4		48 times	
3	UV	60°C	2.5 h	UVA-340 nm
4	Spraying		0.5 h	6–7 L/mn, UV lamps switched off
5	Go to step 1			
		Total (1 cycle) = 168 h		

properties of these systems applied onto wood samples when they were exposed to artificial ageing. Improvements to the durability of clear finish-wood systems coupled to ease of application would help to increase demand for wood, which is loosing market share to more durable materials such as aluminum and PVC in industrial joinery. The durability of finished wood can be predicted or evaluated using different macroscopic methods such as the measurement of contact angle and water absoption,[20] the determination of glass transition temperature during weathering,[21] the measurement of color change expressed using the CIE L*a*b*system,[22–24] the amount of cracks that appear at the samples surface. At the molecular scale, IR spectroscopy can also be used and has been applied to characterize materials before and after natural or artificial ageing[25,26] and to explain UV-photochemical degradation of polymers.[27] In this study, we exposed specimens to accelerated ageing and assessed the durability using the techniques mentioned above.

MATERIALS AND METHODS

Two wood species were selected which contained low and high concentrations of extractives that can interfere with the curing of UV-curable finishes. Spruce (*Picea abies*) was selected because it contains only a few percent of extractives. Conversely oak (*Quercus robur*) was selected because it contains a very high content of extractives (mainly quercetine and ellagitanins). Both species have been used for outdoor joinery.

The dimensions of wood samples for testing were $100 \times 60 \times 6$ mm^3 (orientations longitudinal-tangential).

The resins used in this study were acrylate oligomers supplied by UCB Chemicals. Their trade names are: Ebecryl 8402 (aliphatic polyurethane acrylate) and Ebecryl 605 (epoxy diacrylate) respectively, diluted by 50 and 30% with 1,6-hexanediol diacrylate HDDA.

Phenyl bis(2,4,6-trimethylbenzoyl)-phosphine oxide BAPO (97%), isopropylthioxanthone ITX (97%), and *N*-methyldiethanolamine MDEA (>99%) were used together as initiating system and their quantities were fixed at 0.2; 0.2 and 0.5% weight/resin,

respectively. They were purchased from ALDRICH and used as received without further purification.

These photoinitiators have an absorption band that extends into the visible region of the electromagnetic spectrum and thus can initiate polymerization under visible light.[13,18]

Two different organic and inorganic UV absorbers were tested. The UV absorber Tinuvin 1130 is an organic compound based on 2-hydroxyphenyl triazole which was provided by Ciba Specialty Chemicals Inc. Inorganic absorber Rhodigard S100 was furnished by Rhodia Electronics and Catalysis as a nanodispersion of cerium oxide with a diameter of particles comprised between 5 and 100 nm. The quantities of Tinuvin 1130 and Rhodigard S100 were fixed at 3 and 2.5% weight/resin respectively, according to the suppliers' specifications.

The formulations were prepared and applied to wood samples by brushing. They were then dried in a UV bench fitted with four Heraeus Amba lamps (20 mm diameter and length of the arc 195 mm) i.e., two mercury vapor lamps (emission wavelengths : 313, 364, 405, 436 nm) and two metal-halide (gallium) (emission wavelengths : 403, 417 nm). The curing was extended until the finish was "tack free." The thickness of dry film was evaluated to 100 μm. The ageing test was carried out in a QUV weatherometer (Q-Panel society). The irradiation system was constituted of 8 lamps of UVA-340 nm type (i.e., lighting of 0.68 W/m^2). A water spraying system was added to the original device.

An artificial weathering cycle was used which simulates outdoor conditions. This cycle is described in Table I.[28] The durability of the coatings was evaluated using different techniques. The variation of color was measured in the CIE L*a*b* system using a spectrocolorimeter Spectro-color (Dr Lange Gmbh) with a standard illuminant D65 and 10° circular illumination. Mean values of brightness L* and chromatic coordinates a* and b* were calculated from ten measurements at different positions on each sample. Measuring area was 0.8 cm^2. Color variations as a result of weathering are expressed as the overall color difference ΔE^* given in the CIE *L*a*b** system by $\Delta E^* = [\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}]^{1/2}$ and correlated to chroma $C^* = (a^{*2} + b^{*2})^{1/2}$ and hue angle $h^* = \arctan (b^*/a^*)$.
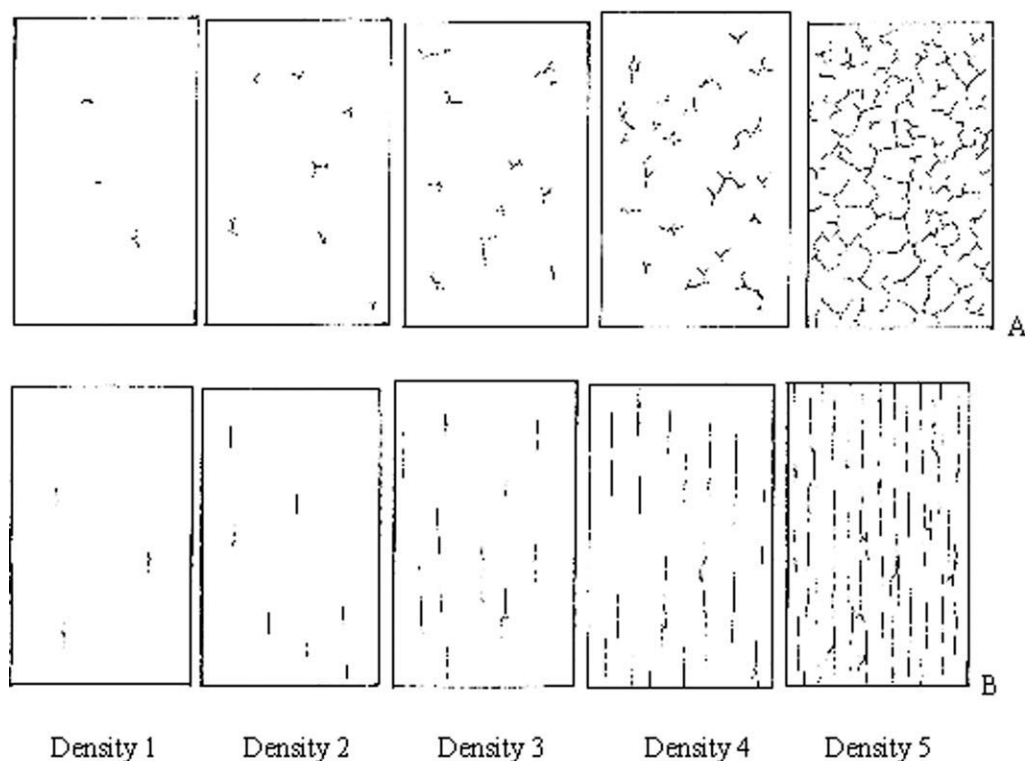
Rating	Quantity of cracks
1	Very few, i.e. small, barely significant number of cracks
2	Few, i.e. small but significant number of cracks
3	Moderate number of cracks
4	Considerable number of cracks
5	Dense pattern of cracks

Figure 1 Subjective evaluation of cracks on panels area from 1 to 2 dm^2 according ISO4628-4 : 2003(E) : (A) without preferential direction and (B) with one preferential direction (for example due to brush marks or wood grain). Correspondence between density and quantity of cracks.

The degradation of clear finishes/wood systems can be estimated by quantifying the cracks that appear at the surface. This quantification was done from a visual control of samples according to the specifications of ISO 4628/4 : 2003(E)[29] and by referring to the correspondence table given in Figure 1.

IR spectra of finishes applied onto wood and glass (control) before and after accelerated ageing were recorded in Attenuated Total Reflection mode using an IR microscope AutoIMAGE Perkin–Elmer. This device is a Fourier Transform InfraRed spectrophotometer (Spectrum 100 Perkin–Elmer) coupled to a microscope controlled by a computer. The photovoltaic detector consists of a mercury-cadmium-tellurium alloy and has high sensitivity, which can be enhanced by cooling with liquid nitrogen.

The range between 4000 and 400 cm^{-1} was scanned 50 times for each spectrum and each sample was analyzed three times.
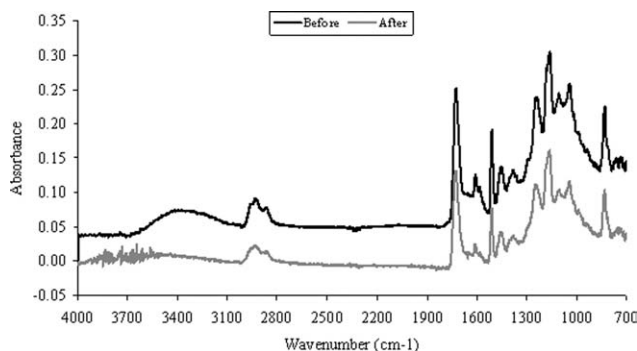
RESULTS AND DISCUSSION

Characterization of films by FTIR microscopy

The results obtained with the epoxy acrylate resin are discussed first. Figure 2 shows the IR spectra of the unweathered epoxy acrylate resin without UV absorbers on oak, spruce, and glass. It appears that
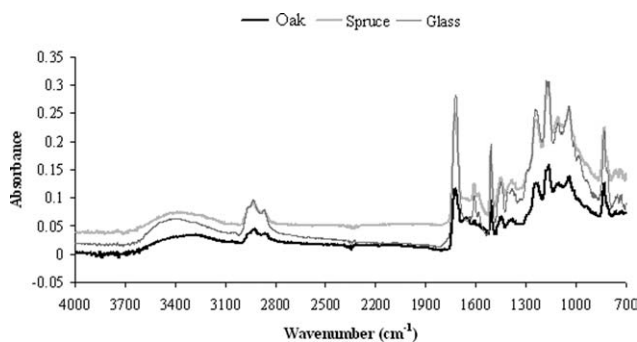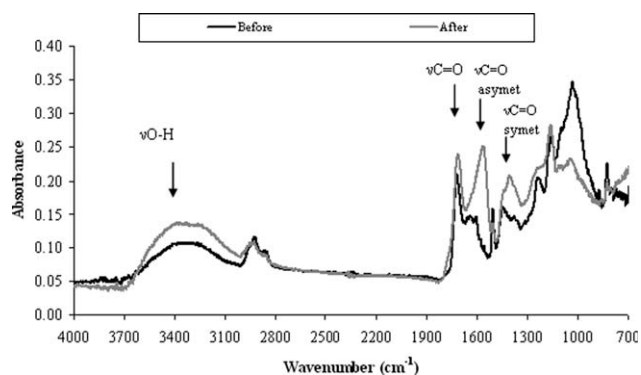
Figure 2 IR spectra of Epoxy acrylate applied on spruce and oak before ageing (glass substrate used as reference).

there is no significant difference between spectra whatever the substrate used. Thus the presence of extractives in oak does not seem to influence the nature of the polymer formed even though previous research has shown that they can inhibit the curing of UV finishes.[30] This finding is not so surprising if we consider the analysis depth by IR spectroscopy in ATR mode i.e., 1–2 µm which is only representative of the surface and not of the film in the bulk (film thickness about 100 µm). It would be interesting to implement other characterization techniques (Raman spectroscopy or IR spectroscopy in transmission mode on free finish film) to observe potential polymerization gradients. The characteristic bands of epoxy and acrylate groups are present in spectra i.e., νC=O at 1720 cm^{-1}, νO—H at 3400 cm^{-1} and νC—H at 2900 cm^{-1}. After accelerated ageing, the spectra (intensity and position of bands) are largely unchanged as shown for example in Figure 3 for the resin applied onto spruce. In contrast, as soon as UV absorbers are incorporated into the formulation, some noticeable differences are observed in spectra before and after ageing. When Tinuvin 1130 is added to the coatings (Fig. 4), we observe a degradation of the acrylate after ageing. This degradation is possibly due to a hydrolysis of the acrylate functions into carboxylates which are able to migrate towards the surface as suggested by the appearance of two
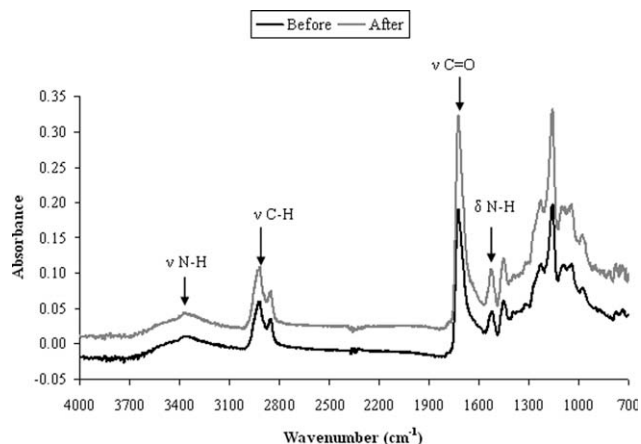

Figure 4 IR spectra of Epoxy acrylate in presence of Tinuvin 1130 applied on spruce before and after ageing.

bands at 1560 and 1390 cm^{-1} corresponding respectively, to the asymmetric and symmetric stretching bands for carboxylates groups. Besides, the superimposition of the tail of the νC=O asymmetric band with νC=O of epoxy acrylate seems to enhance the νC=O band at 1720 cm^{-1} whereas this band could be almost unchanged or slightly lower. The degradation of acrylate functions into alcohols can also be assumed as shown by an increase of the νO—H band at 3400 cm^{-1}. The modification of the νO—H band could also originate from the presence of water at the film surface, the hydrophilic character of which is assumed to increase with ageing time. With the UV absorber Rhodigard S 100, we have observed the same trends i.e., the variations of the bands were of the same order and confirm the degradation of the acrylate. These findings indicate that the films based on epoxy acrylate are less susceptible to photodegradation when they do not contain the tested photostabilizers.

The results obtained from the urethane acrylate resin are discussed now. Figure 5 shows IR spectra of the urethane acrylate resin without UV absorbers on oak before and after accelerated ageing. As

Figure 3 IR spectra of Epoxy acrylate applied on spruce before and after ageing.
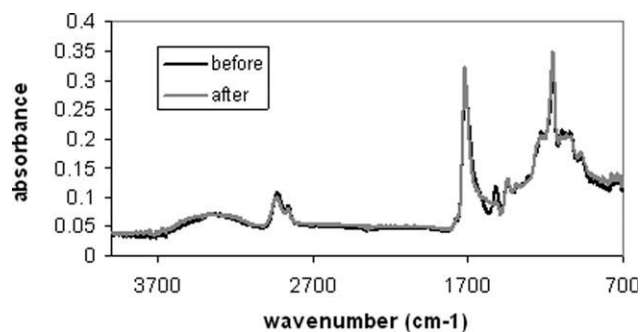

Figure 5 IR spectra of urethane acrylate applied on oak before and after ageing.
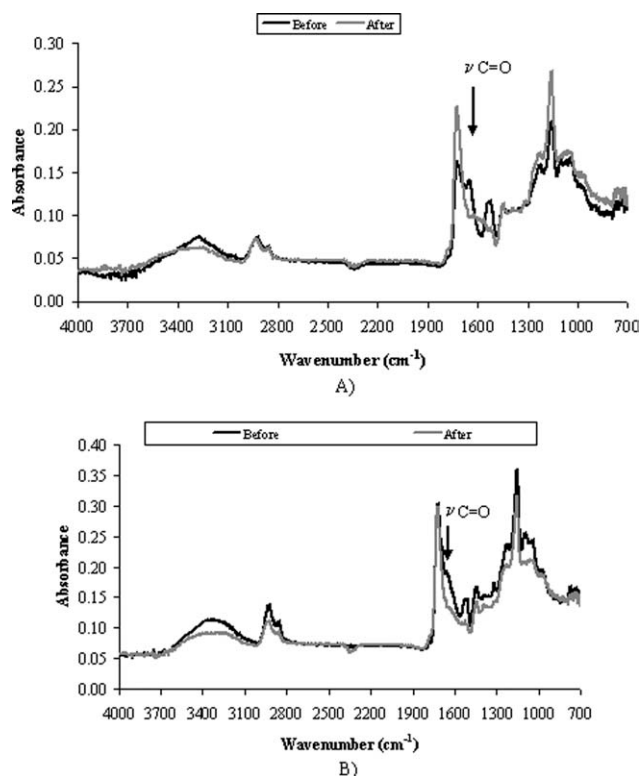
Figure 6 IR spectra of urethane acrylate in presence of (A) Tinuvin 1130 and (B) Rhodigard S100 applied on oak before and after ageing.

Figure 7 IR spectra of urethane acrylate in presence of tinuvin 1130 applied on spruce before and after ageing.

previously, a film applied onto glass was used as the control (results not presented here) and its spectrum did not reveal significant difference before ageing compared to film cured onto wood. The characteristic elongation and deformation bands of Ebecryl 8402 can be observed i.e., $\nu N{-}H$ around 3000–3500 cm^{-1} for urethanes, $\nu C{-}H$ around 2800–2900 cm^{-1} for alkyls, $\nu C{=}O$ in the range 1600–1700 cm^{-1} of acrylates and δN-H at 1512 cm^{-1} of amides (Fig. 5). The ageing process does not greatly influence the intensity and position of these absorption bands. On the contrary, the introduction of UV absorbers leads to large modification in the spectra of films as shown in Figure 6. Before ageing, additional peaks are present

at 1644 cm^{-1} and 1650 cm^{-1} for the systems containing Tinuvin 1130 and Rhodigard S100 respectively. If we assume that this elongation vibration $\nu C{=}O$ is typical of urethane groups,[31,32] this result seems to indicate that polymerization of the film in the presence of UV absorbers does not lead to the same film as that cured in absence of UV absorbers. The film surface appears enriched by urethane groups probably due to a surface reorganization of urethane versus acrylate functions.

But, by taking into account the high extractives content in oak, we could also assign the 1640–1650 cm^{-1} band to some wood phenolic substances which have migrated towards surface.

After ageing, for the samples containing Tinuvin 1130, the intensity of $\nu C{-}H$ is almost unchanged whereas $\delta N{-}H$, $\nu N{-}H$ and $\nu C{=}O$ (at 1644 cm^{-1}) bands decrease significantly. We conclude that there was a preferential degradation of urethane at the film surface. The C—O band at 1720 cm^{-1} appears higher after ageing because of the higher part of the acrylate functions comparatively to the one of urethane groups.

Note that the decrease of $\nu C{=}O$ at 1644 cm^{-1} could also result from a degradation of the extractives which have migrated and concentrated at the surface as previously mentioned.

For the samples containing Rhodigard S100, the intensity of $\delta N{-}H$, $\nu N{-}H$, and $\nu C{=}O$ (at 1650 cm^{-1})

TABLE II
Evaluation of the Cracks Appearing at the Surface of Acrylate Films During the Ageing Process (the Notation Scale Ranges from 0 (No Cracks Visible on Surface) to 5 (Large Defects Cover the Overall Surface)

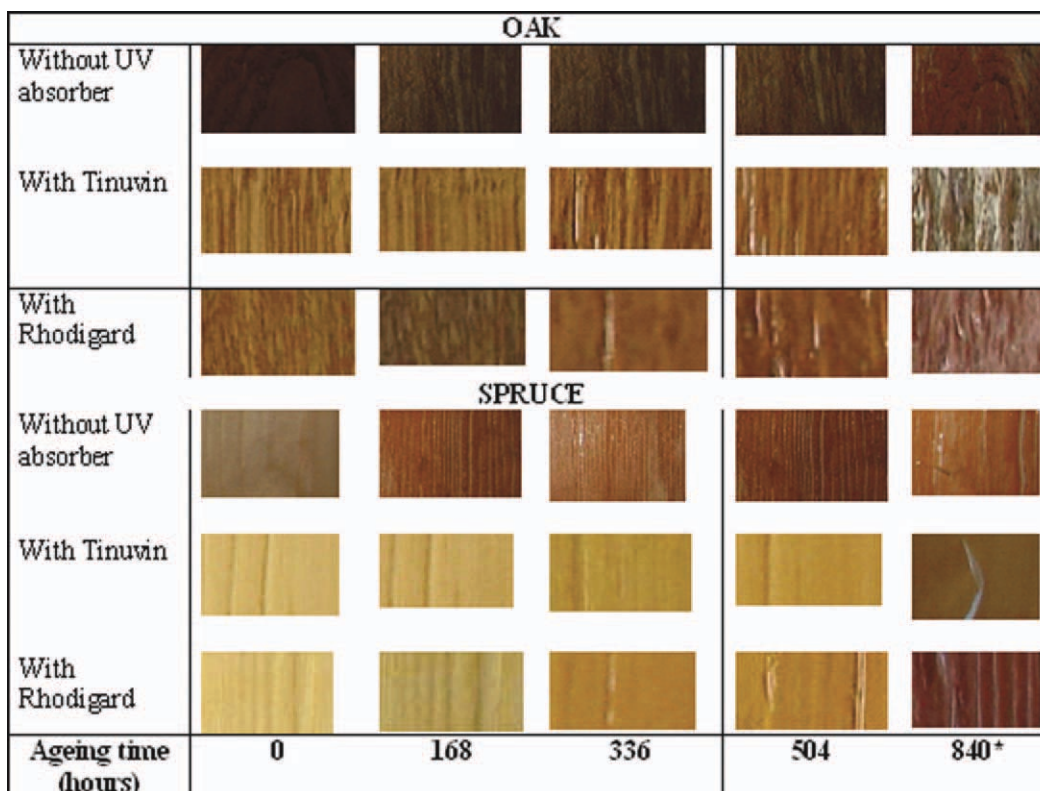|		Spruce					Oak				
		Ageing time (h)					Ageing time (h)				
Initiating system :BAPO/ITX/MDEA		168	336	504	672	840	168	336	504	672	840
Epoxy acrylate+30%HDDA	None	1	3	5	5	5	3	5	5	5	5
	Tinuvin 1130	0	0	0	1	3	0	1	2	3	5
	Rhodigard S100	0	0	1	4	5	0	2	3	4	5
Urethane acrylate +50%HDDA	None	2	5	5	5	5	4	5	5	5	5
	Tinuvin 1130	2	4	5	5	5	3	5	5	5	5
	Rhodigard S100	2	5	5	5	5	2	5	5	5	5

Figure 8 Ageing effect on epoxy acrylate film applied on oak and spruce (*only 672 h of ageing for system without absorber). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
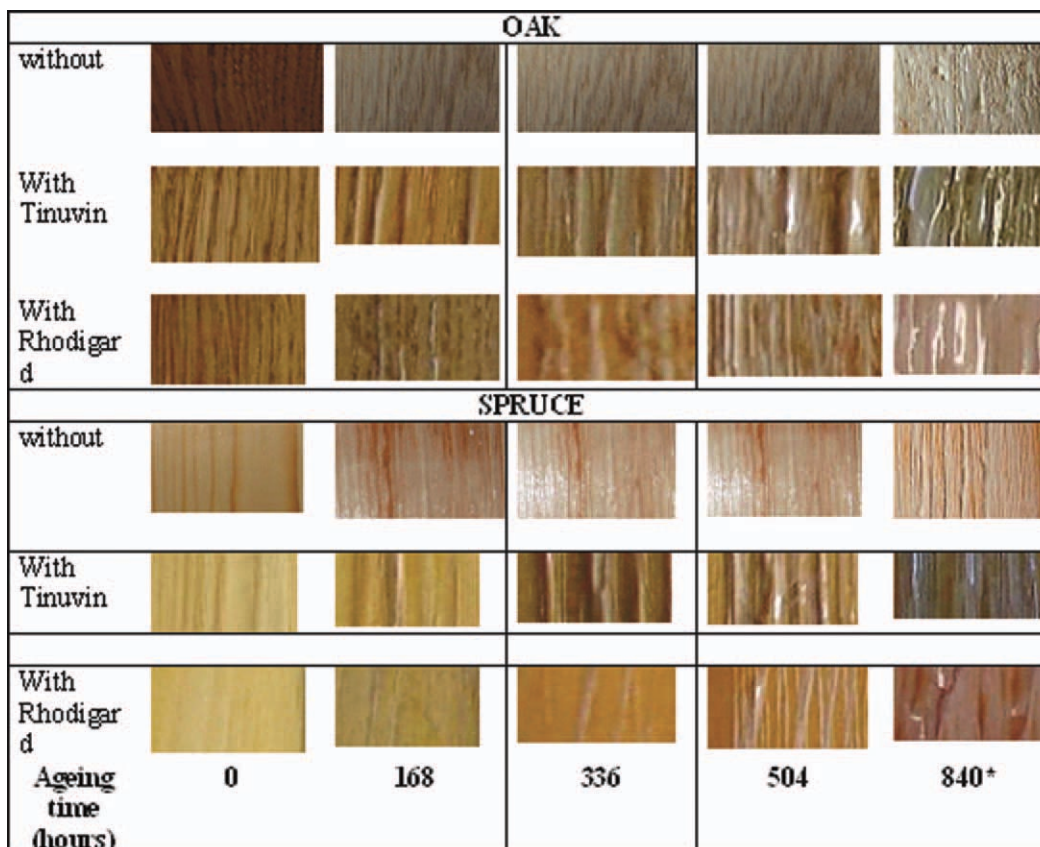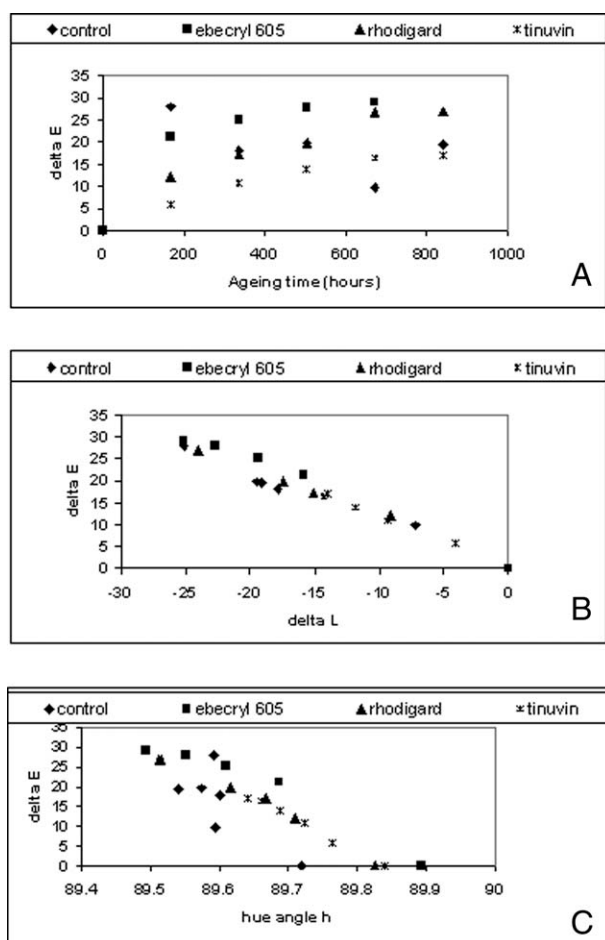

Figure 9 Ageing effect on urethane acrylate film applied on oak and spruce (*only 672 h of ageing for system without absorber). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 10 Color modifications for epoxy acrylate film applied on spruce. Color variation as a function of (A) ageing time, (B) brightness variation, (C) hue angle *h*.

bands decreased as well as νC—H to a lesser extent. It is possible that the cerium dioxide CeO_2 has some photocatalytic properties that it could be interesting to characterize as reported in literature.[33,34]

These observations can not be done as urethane acrylate resin is applied onto spruce. The films cured in the presence or absence of UV absorbers and analyzed before ageing are almost equivalent. The elongation band νC=O near 1640 cm^{-1} is not visible indicating no enrichment of the surface by urethane groups according to our first assumption or no migration of extractives towards the surface as proposed in the second one. The lower amount of extractives present in spruce (around 3-5% compared to 5-10% in oak[35]) and their different nature could explain this result. After ageing, the δN—H band around 1510 cm^{-1} decreased as well as the νC—H band in a much less extent but we notice no modification of the νN—H band (Fig. 7). The degradation of the film on spruce is then less pronounced than previously in the case of oak. These findings may be explained by a difference in the permeability of the finish film cured onto oak or spruce towards

water. Knowing that phenolic extractives are able to retard or inhibit polymerization, we expect to have a more homogeneous polymer in the bulk with a higher crosslinked network when the finish is cured on spruce than on oak, hence a less permeable film. We can also assume that the degree of cure is not homogeneous from the surface of the coating to the interface wood/resin. As a matter of fact, some examples of such a problem of cure gradient can be found in literature with other polymeric systems.[36,37] Some experiments are now in progress to check this assumption.

Macroscopic observations of films: Cracks and color modifications

From a macroscopic point of view, the photostabilizers appeared to protect the coatings but not at the same extent and in the same manner for both resins. Table II shows the crack levels of films during accelerated weathering. The effects of ageing are also
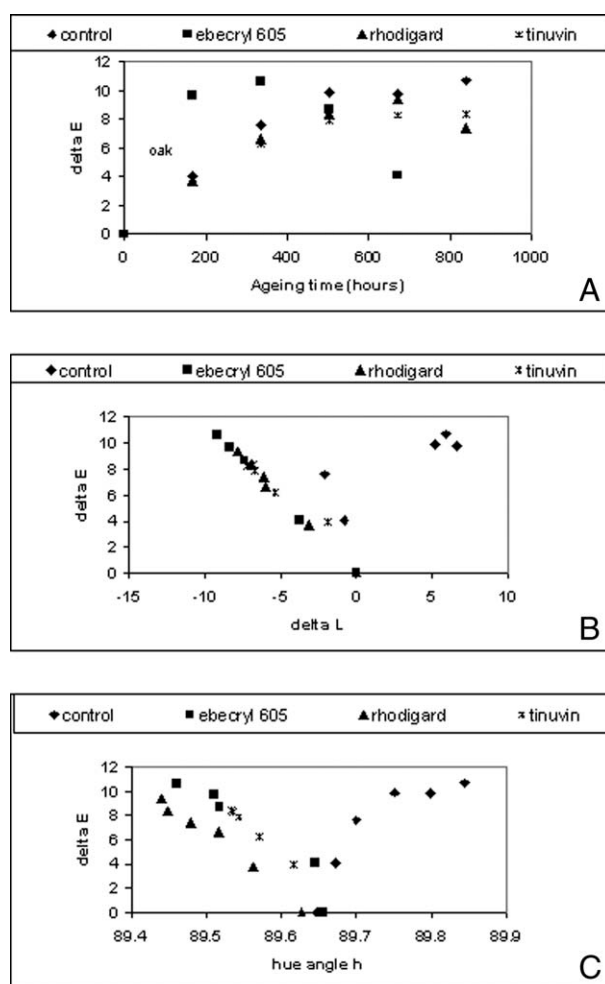
Figure 11 Color modifications for epoxy acrylate film applied on oak. Color variation as a function of (A) ageing time, (B) brightness variation, (C) hue angle *h*.
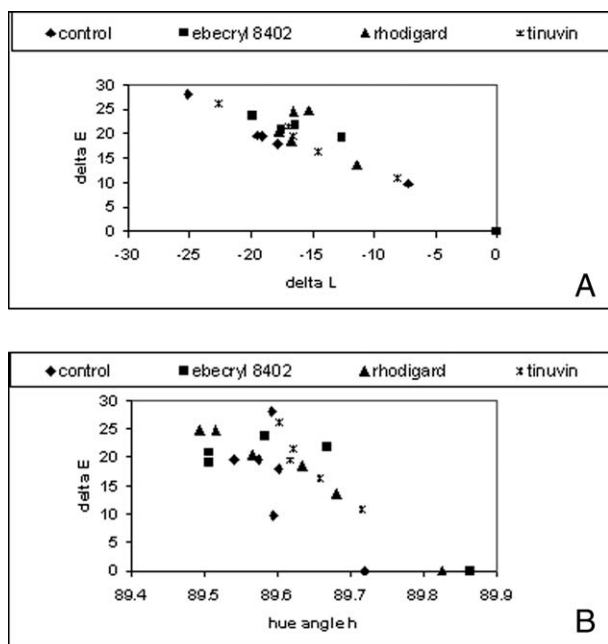
Figure 12 Color modifications for urethane acrylate film applied on spruce. Color variation as a function of (A) brightness variation, (B) hue angle h.

illustrated in Figures 8 and 9. It appears that the addition of UV absorbers into the epoxy acrylate finishes retarded significantly the degradation of films applied to spruce and oak since maximal level of cracking (Level 5) was reached after 504 and 336 h compared with 840 h for the same finishes containing UV absorbers. Tinuvin 1130 appears to be slightly better at reducing cracking than Rhodigard S100 and the degradation of films on spruce are less extended than on oak.

As the urethane acrylate finishes are concerned, the addition of UV absorbers in the formulation retards only slightly the ageing effect of the coating at the beginning of the weathering process (in the first 168 h) but it appears no more efficient for longer times, whatever the nature of wood species and UV absorber.

Figures 10 and 11 show the variation of color (ΔE^*) of the finishes as a function of (a) the ageing time (b) the variation of luminance ΔL^*, and (c) hue angle h^* for epoxy acrylate respectively, applied to spruce or oak whereas Figures 12 and 13 show the results for acrylate urethane.

The case of epoxy acrylate is discussed first. In the case of spruce, modifications of color, which were observed in the first hours of exposure, were very large (it is noteworthy that a variation of one unity can be detected visually) but finally stabilize quickly (10a).[24] From a general point of view, the overall ΔE^* determined for the substrate or the substrate coated with unmodified resin was slightly higher ($\Delta E^* >$ or $= 20$) than those obtained for samples

with the finishes that contained UV absorbers. It appears that Tinuvin is initially better than Rhodigard at restricting color changes in accord with results for cracking. The variation in ΔE^* is associated with variation in ΔL^* (10b). The color variation decreases as brightness one increases. The very slight variation of hue angle h^* (10c) seems to mean that chromophoric species are unmodified during ageing whatever the system investigated. For oak samples (11a), the color variation as function of ageing time was large and UV absorbers did not restrict color changes. Again color modifications ΔE^* were correlated with luminance variations: as soon as finishes were applied to the substrate, the ΔE^* observed decreases as ΔL^* increases, irrespective of whether the finishes contained photostabilizers. For uncoated oak, we observed that wood which was originally dark become lighter as the ageing time increases and in this case ΔE^* increases as ΔL^* increases (11b). The same trend was observed for the curves giving ΔE^* as function of hue angle h^* even if h^* varies only very slightly (11c).

We now comment results concerning urethane acrylate. For spruce samples, the same trends to those obtained with ebecryl 605 were observed i.e., the variation of color is directly correlated to the variation of brightness and decreases as the brightness increases (12a). For oak samples, the graph showing ΔE^* as a function of ΔL^* is different to that obtained with the previous resin insofar as color variations of control and coated samples showed similar behavior. We can also note that ΔE^* values increased as
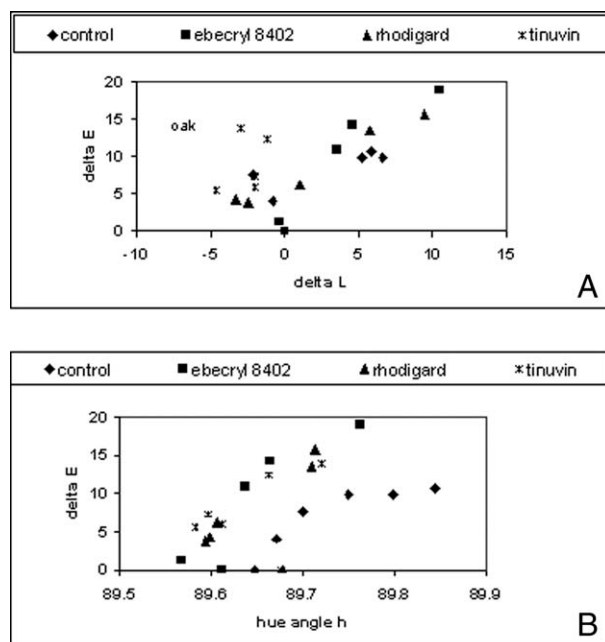
Figure 13 Color modifications for urethane acrylate film applied on oak. Color variation as a function of (A) brightness variation, (B) hue angle h.

the sample became brighter (13a). With both wood species, ΔE^* decreased as hue angle h^* increased (12b and 13b). Again, tested UV absorbers do not seem to have a clear significant protective effect against photodegradation.

CONCLUSIONS

In this study, we attempted to evaluate the effect of an artificial ageing process onto the durability of "clear finishes/wood" systems by implementing different techniques, merely IR spectroscopy, color measurements, and cracks formation.

It appears that the observations made at a molecular scale are not necessary linked to the ones made from a macroscopic point of view. The formation and quantity of cracks onto surface samples is retarded and the color variations are slightly less pronounced when UV absorbers are added to both tested resins. On the contrary and surprisingly, IR spectra show clear differences of the material before and after ageing as soon as UV absorbers are present in the finishes. For example, segregation phenomena and preferential degradation of one part of the polymer were observed (Epoxy more resistant than acrylate in Ebecryl 605 and acrylate > urethane in Ebecryl 8402. The nature of wood species appears also significant onto the durability of the system.

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