THE EFFECTS OF ULTRAVIOLET RADIATION ON POLYCARBONATE GLAZING

Effects of UV on polycarbonate glazing

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Abstract

Unstabilised bisphenol A polycarbonate samples were aged under UV radiation for periods of up to 3000 hours. The samples were then characterised using FTIR/ATR, GPC, SEM, microscopy, light transmission and colour changes. The changes in optical properties were related back to the changes in the polymer microstructure and composition.

In this study, the molecular weight of polycarbonate was seen to decrease during UV exposure, making it susceptible to embrittlement and surface erosion. This coupled with the discoloration of the polymer contributed to the loss of optical properties, including reduced light transmission, loss of gloss, increased haze, and discoloration. In the case of reduced light transmission and yellowing of polycarbonate, the degradation mechanisms were identified through infra-red spectroscopy. Photo-induced yellowing was accelerated by humidity, while the reduction in other optical properties was more severe in low humidity environments.

Keywords: Polycarbonate, photodegradation, photo-oxidation, molecular changes, physical characterisation, FTIR/ATR, surface analysis

1 Introduction

Polycarbonates are a very special class of engineering thermoplastic. They exhibit a unique combination of properties, for instance they have excellent toughness compared to other thermoplastics, have high transparency and dimensional stability over a wide temperature range, have good electrical properties and are lightweight. As a consequence, polycarbonates have established their niche in a wide range of applications, including building and construction where they are often used as a lighter and tougher substitute for glass or metal, e.g. roofing and glazing.

However, bisphenol A polycarbonate is susceptible to photodegradation under ultraviolet (UV) radiation, which causes breakdown of the material, leading to yellowing, loss of toughness and embrittlement. In this paper, the authors look into the effects of UV on polycarbonate degradation, with the aim of understanding some of the relationships between chemical and physical changes taking place in polycarbonate and how the overall performance of the material is affected.

1.1 The effect of UV

When sunlight falls onto polycarbonate, the polycarbonate molecules absorb energy of certain wavelengths in the UV range, this is particularly so for the ester groups and the aromatic rings which are strong UV absorbers. The absorbed energy can cause the rupture of covalent bonds initiating photo-oxidation and photo-Fries reactions (Factor 1996).

Both mechanisms are distinct, i.e. they follow their own routes and have distinct intermediates and reaction products. The mechanisms are also wavelength dependent, i.e. one reaction predominates over the other depending on the wavelength of the incident radiation (Andrady et al. 1992). Photo-Fries is triggered by λ <310 nm and photo-oxidation is favoured by wavelengths in the 290–350 nm range. In the environment, both mechanisms can take place and compete with each other (Rivaton 1995).

1.2 Photo-fries

The photo-Fries reaction is promoted by radiation of short wavelengths and does not require oxygen to occur. In the photo-Fries mechanism, the energy absorbed from UV radiation promotes the scission of the carbonate linkage, forming two primary free radicals (Rivaton 1995). The free radicals then rearrange to form phenyl salicylates and dihydroxybenzophenones, and other groups such as dihydroxybiphenyl and hydroxydiphenyl ether groups. Oxidation of these groups results in substances such as ortho-dihydroxybenzophenone or diphenoquinone which absorb in the blue range, and are responsible for the yellowing observed in photodegraded samples (Andrady et al. 1992; Clark and Munro 1984; Pryde 1984). The reaction mechanism is shown in Figure 1.

The products formed during photo-Fries reactions can also be photo-oxidised, yielding secondary and tertiary products of lower molecular weight.

1.3 Photo-oxidation

For samples exposed to sunlight and air, photo-oxidation has been reported to be the predominant reaction, which occurs via a three-stage reaction (Clark and Munro 1982; 1984; Pryde 1984). In photo-oxidation (shown in Figure 2), the UV radiation is absorbed by impurities in the polymer, and as a result hydrogen atoms are abstracted from the methyl groups, forming a free radical and thus initiating a chain scission reaction which propagates the reaction. In the presence of oxygen, the methyl side chains are photo-oxidised into hydroperoxide intermediates, which are transformed into tertiary alcohols and ketones. Further photo-oxidation of the products and of the aromatic rings also contributes to the yellowing of the polymer. Moreover, the free radical reaction is self-propagating until no more active sites are available or no oxygen is present to further the reaction.

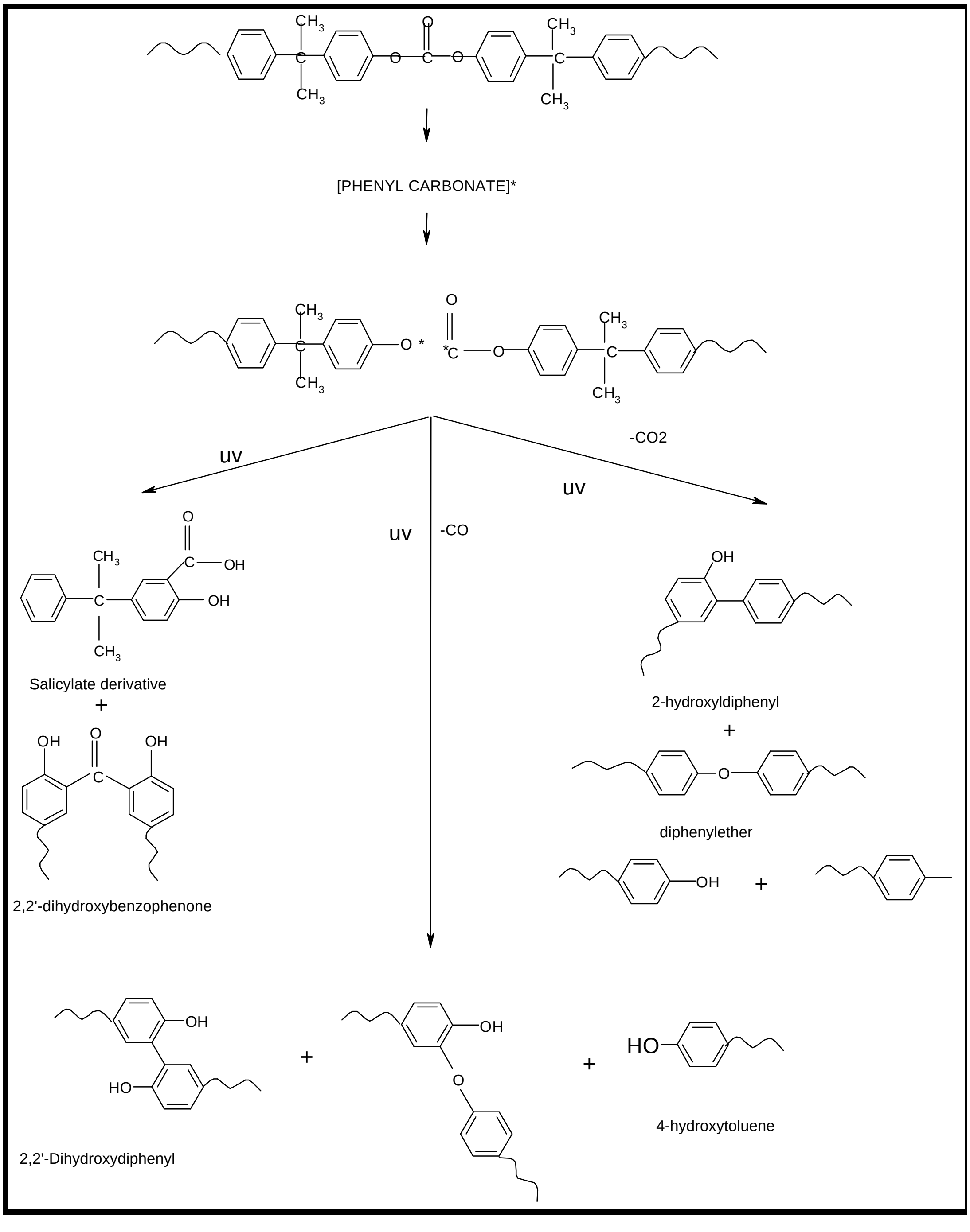


Fig. 1: Photo-fries reaction (source: Rivaton 1995)

The photo-oxidation of the aromatic rings has also been discussed to be a viable cause of yellowness, according to a study of model compounds, such as m-

BPO (3,3’-dihydroxy-diphenyl ether). When exposed to UV radiation under artificial and natural weathering conditions, these compounds showed yellowness index changes (Factor et al. 1987).

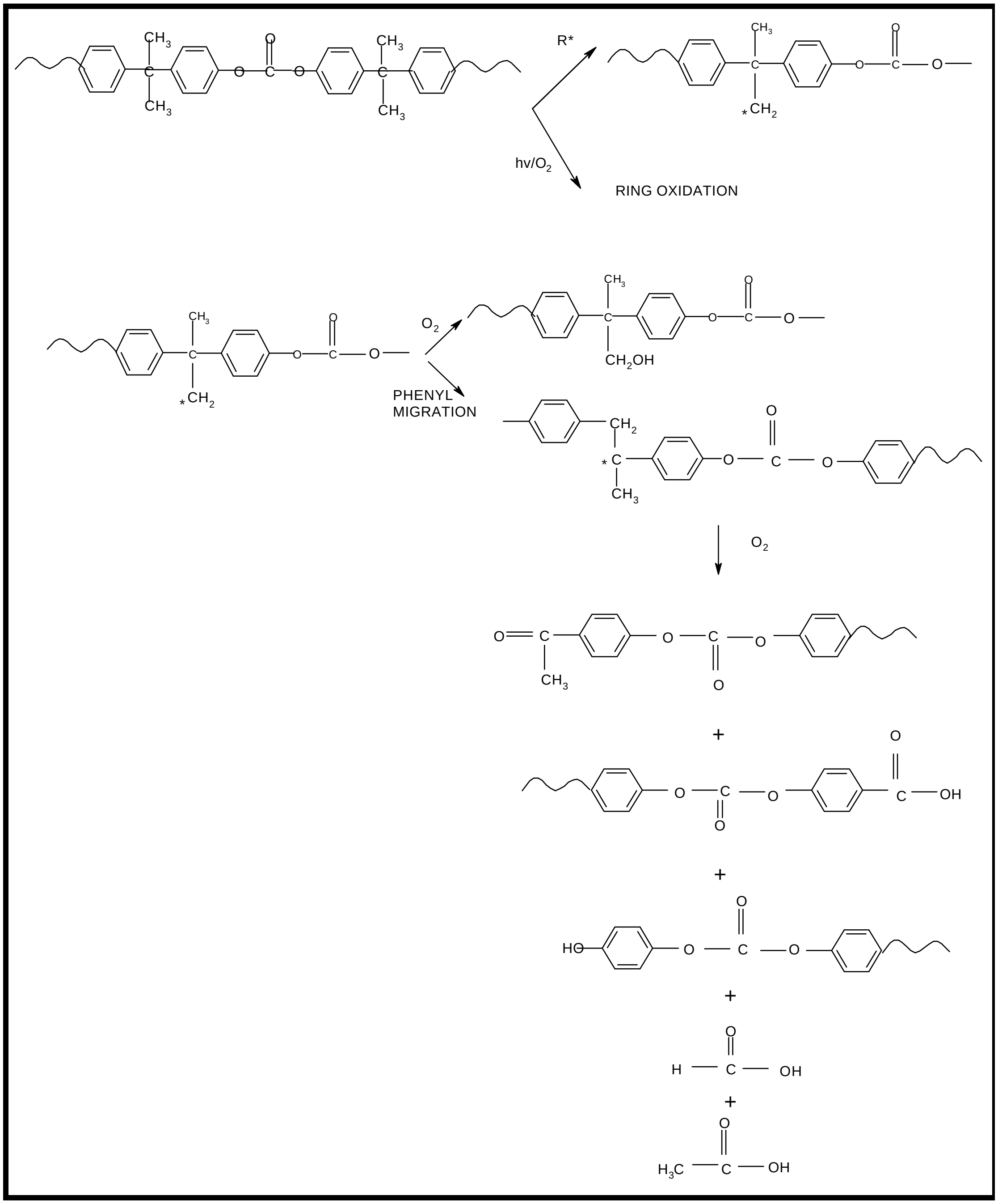


Fig. 2: Photo-oxidation of polycarbonate (source: Rivaton 1995)

In photo-oxidation, chain scission is favoured over cross-linking, particularly near polymer chain ends resulting in the formation of low molecular weight compounds. At short wavelengths, degradation free radicals tend to recombine into products, while under long wavelengths they react separately forming structures with phenolic end-groups (Dilks 1989; Gorelov and Miller 1978).

Factor and Chu (1980) identified dual mechanisms acting in polycarbonate degradation. They noted that after irradiation of polymer films, two layers were formed – a superficial layer, brown and strongly oxidised, and beneath it a yellow layer. The superficial layer was attributed to photo-oxidation by short wavelengths, because the outer skin of the polymer would absorb the shorter wavelengths. Longer wavelengths would penetrate further into the polymer and promote the formation of the second layer. The degradation mechanism could thus be explained by a combination of photo-Fries followed by photo-oxidation at the surface layer caused by short wavelengths (<310 nm) and photo-oxidation caused by longer wavelengths in the inner layer. Hoyle et al. (1993) and Rivaton (1995) have verified that photo-Fries products could act as sites for photo-oxidation and be destroyed in the process, which would explain why only photo-oxidation and no photo-Fries products were identified in the polymer after long exposure periods.

1. 4 Photobleaching

Degradation products that form from the exposure of polycarbonate to UV radiation can favour or hinder reactions that occur with further exposure, e.g. phenyl salicylate was reported to act as a photosensitiser, while dihydroxybenzophenone inhibits photo-oxidation (Rivaton 1995).

Rivaton (1995) reported that when yellow photolytic species were formed at very low concentration they could photo-induce the radical oxygenation of polycarbonate. On the other hand, as the concentration increased, these coloured species could also shield the polymer by decreasing the penetration of longer wavelength UV (λ >310 nm) into the inner polymer layers, thus reducing photo-oxidation. Further, photo-oxidation of the degradation products could also contribute to the destruction of coloured species and the formation of smaller polar molecules – a process known as photobleaching.

Andrady et al. (1992) reported the occurrence of photobleaching as the photodegraded polymer was exposed to longer wavelengths (λ ~340 nm). Photobleaching is in general observed after extensive exposure, when the rate of destruction of coloured species outgrows their rate of formation. However, it does not restore the damage caused by degradation.

1.5 Contribution of humidity

A review of the literature on the effect of humidity on polycarbonate discoloration has uncovered a number of conflicting observations. Polycarbonate is generally known to be inert to water at ambient conditions. It only undergoes hydrolysis under high pH (basic) conditions coupled with elevated temperatures. In such cases, the reverse of the condensation mechanism occurs and the polymer reverts to its original constituents. Under normal atmospheric conditions, the low reactivity of polycarbonate is attributed to the absence of polar groups in the molecule.

When photo-induced reactions under UV radiation occur, new degradation products are formed, and their reactivity to water could affect the polymer’s susceptibility to UV. Some researchers have observed that humidity accelerates the process of photo-yellowing of polycarbonate (Rivaton et al. 1986).

Conversely, Factor and Chu (1980) and Pryde (1985) observed that high humidity levels slowed the yellowing process. Pryde (1985) observed that for samples exposed to λ >284 nm, relative humidity retarded the onset of degradation. This effect was more easily perceptible in samples irradiated in nitrogen where the controlling mechanism would have been photo-Fries. Pryde attributed the decreased reaction rate to the reaction between water and the excited species formed by photo-Fries. The hydrogen in water would be abstracted by the excited species and react with it, thus preventing the propagation of the reaction in the polymer molecule, decreasing the initial scission to radicals.

Rivaton et al. (1986) argued that chain scission could favour the hydrolysis of the polymer during exposure to water for long periods of time. Although the photo-oxidation (chain scission) products do not react directly with water, the chain breaking resulted in the formation of polar groups that favoured hydrolysis, releasing monomers and oligomers, which offer less resistance to oxidation compared to the original polycarbonate units (Rivaton et al. 1986). Thus the photo-oxidation rate is increased in the presence of high humidity levels. This would explain the observation by Factor and Chu (1980) that the exposed surface is more susceptible to hydrolysis than the bulk of the polymer.

In contrast to the effect of humidity on polymer degradation, several authors have discussed the solubility of the degraded species. Pryde (1984) suggested that the onset of photodegradation via a photo-Fries reaction (short wavelength) yields products which are hydrophobic and thus these products would not react directly with water. In contrast, for photo-oxidation at short wavelengths, as discussed by Clark and Munro (1983) polar products were formed that could be dissolved in water and leached out of the polymer. This would suggest that photo-Fries reactions have not occurred in this case, or that if they did occur, further photo-oxidation reactions occurred to convert the photo-Fries products to soluble products.

2 Experimental

Evaluation of the effect of UV radiation on polycarbonate sheets was achieved by aging the samples using an air-rich environment and a source of UV that simulated the sunlight spectra. Commercial Lexan® bisphenol A polycarbonate film, manufactured by GE Plastics, was used in this work. The samples were 1 mm thick and unstabilised (Mw ~60,350; Mn ~32,300).

The samples were exposed in a QUV Panel with UVA-340 nm fluorescent lamps. The emission spectra is very similar to that of sunlight for λ <340 nm. The temperature was set at 40 ± 2°C and the relative humidity was either 0 or 42 ± 5%. UV exposure was continuous for intervals up to 3000 hours.

Infra-red analysis was performed in attenuated total reflectance mode (ATR) using a Perkin Elmer 2000 FTIR, with microscope and a germanium tip crystal. The micro-ATR was adequate for measuring the external degraded layer without interference on the spectrum from the bulk polymer.

Surface analysis was performed using a scanning electron microscope (JEOL 35CF), source voltage 25 kV. The samples were coated in gold using sputtering conditions of 18 mA for 60 s. Microscopy was performed using a confocal laser microscope.

The molecular weight was determined by dissolving the samples in tetrahydrofuran AR grade to obtain a 1%w/v solution. The solution was prefiltered (pore size 0.5 μm) prior to analysis. The analysis was conducted in a Waters ALC 26 Gel Permeation Chromatograph (GPC) using narrow polystyrene standards and an injection rate of 1 mL/min.

Light transmission and haze were measured with a Gardner UX-10 Hazemeter, according to AS/NZ 4257.7 (Standards Association of Australia 1987) and ASTM D 1003 (American Society for Testing and Materials 1997a). Gloss was measured with a Gardner BYK Tri Gloss Meter at a 60° angle (American Society for Testing and Materials 1997b).

Discoloration was measured on specimens that were conditioned at 23 $\pm$ 2°C and 50 $\pm$ 5% relative humidity for a period of 48 h prior to each test. The yellowness index (YI) of the samples was measured with a Minolta Chroma Meter CR-200, according to ASTM D 1925-70 (American Society for Testing and Materials 1970)

3 Results and discussion

3.1 Effect of UV on molecular level

In both photo-Fries and photo-oxidation degradation there are three main groups in the polycarbonate molecule that could act as initiation sites, viz.:

- Carbonate groups (photo-Fries reaction).
- Gem-dimethyl group (photo-oxidation).
- Aromatic rings (ring oxidation).

As the degradation progresses, it is expected that photo-oxidation of the primary and secondary products will occur and thus photo-oxidation will predominate, accompanied by chain scission and the formation of low molecular weight compounds. Thus, it is possible to monitor such alterations through determination of functional groups and molecular weight.

Figure 3 shows the ATR spectra of polycarbonate specimens aged for different time periods under UV. In this figure, a broad peak at 3200 cm^{-1} appeared upon exposure to UV radiation. Such a peak indicates the presence of bonded hydroxyl (e.g. in carboxylic acids). As exposure proceeded, broad peaks at 2900 and 2600 cm^{-1} were formed, masking the absorption of the aromatic species. The carbonate peak (1767 cm^{-1}) decreased with exposure. At the same time, the absorption in the carbonyl region (1760–1550 cm^{-1}) increased with new peaks formed at 1701, 1685, 1596 and 1431 cm^{-1}. This is evidence for the formation of a range of photo-oxidation products, with substituted phenols, aromatic esters, aromatic aldehydes, heteroaromatic structures, carboxylic acids and aliphatic esters (1740 cm^{-1}) absorbing in this region.

The decrease in the carbonate peak at 1767 cm^{-1} is a sign that photo-Fries reactions took place, because carbonate sites act as initiation sites for these reactions. The gem-dimethyl peaks at 1390, 1380 and 1360 cm^{-1} were overtaken by the broadening of the carbonyl peaks (1500–1780 cm^{-1}), although there does not seem to be a significant reduction in the concentration of the gem-dimethyl groups. Thus, it is unlikely that the dimethyl groups were the main initiation sites for the reaction. However, we cannot exclude the possibility that a few gem-dimethyl groups might also have served as initiation sites during photo-oxidation reactions. Ring oxidation of aromatic rings is also a dominant reaction because the absorption in the 900–800 cm^{-1} (aromatic C-H) region decreased, indicating an increase in substituted phenolic species.

In summary, the spectra obtained support the affirmations of Rivaton (1995) that UV degradation was initiated by photo-Fries reactions and that as the degradation progresses these reactions are superseded by photo-oxidation reactions. Additional evidence of this is provided by the GPC measurements discussed in the following paragraphs. However, further confirmation of the specific reaction route details is still under investigation.

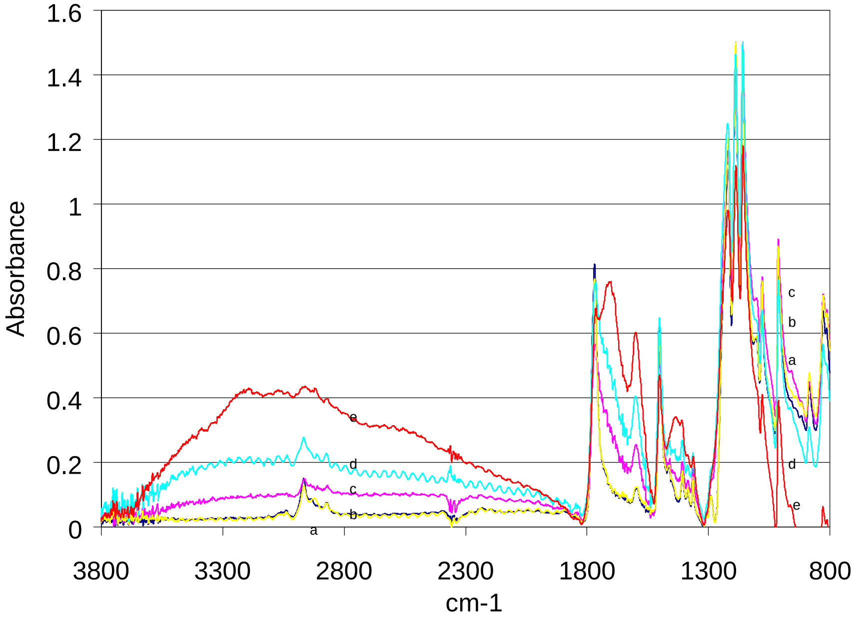


Fig. 3: ATR spectra of polycarbonate exposed to UV at 40°C, 42% relative humidity: (a) control (0 h); (b) 528 h; (c) 1320 h; (d) 1680 h and (e) 2352 h

The molecular weight measurements of polycarbonate exposed to UV radiation indicate the predominance of chain scission rather than cross-linking during the consequent photo-oxidation. Cross-linking is a characteristic of photo-Fries and a large polydispersity (Mw/Mn) ratio denotes its predominance. Thus, if cross-linking predominates there is an increase in the Mw/Mn value. In Figure 4 the average molecular weight and the number average of the samples decreased upon UV exposure, indicating a shift of the molecular weight distribution into lower molecular weight values. For instance, after 2000 h of UV exposure at 0% relative humidity, Mw decreased by 8.9% and Mn decreased by 10.9%, and after 3050 h at 42% relative humidity, Mw was reduced by 3.2%, while Mn was reduced by 6.0%, indicating the occurrence of chain scission in the polymer. The changes were more severe for the 0% relative humidity sample, as discussed later.

Overall, the polydispersity values remained around 2, while in the case of cross-linking, polydispersity would have been expected to increase significantly.

The changes detected in this work were not as dramatic as those of other authors. For example, Sherman et al. (1982) measured a 30% reduction in Mw over 2000 h, but this can be accounted for by the fact that their samples were 0.2–0.3 mm thick, while the samples in our study were 1.0 mm thick. Thus, the overall degradation of their samples would have been more uniform. Chain scission and the formation of smaller molecules would lead to the embrittlement of the polymer. Thus, the surface would be prone to erosion, possible craze initiation, embrittlement and crack propagation. After exposure, some samples were cut with shears, and it was observed that as the time of UV aging increased, the ductility of the polymer decreased and a broader network of cracks extended into the polymer.

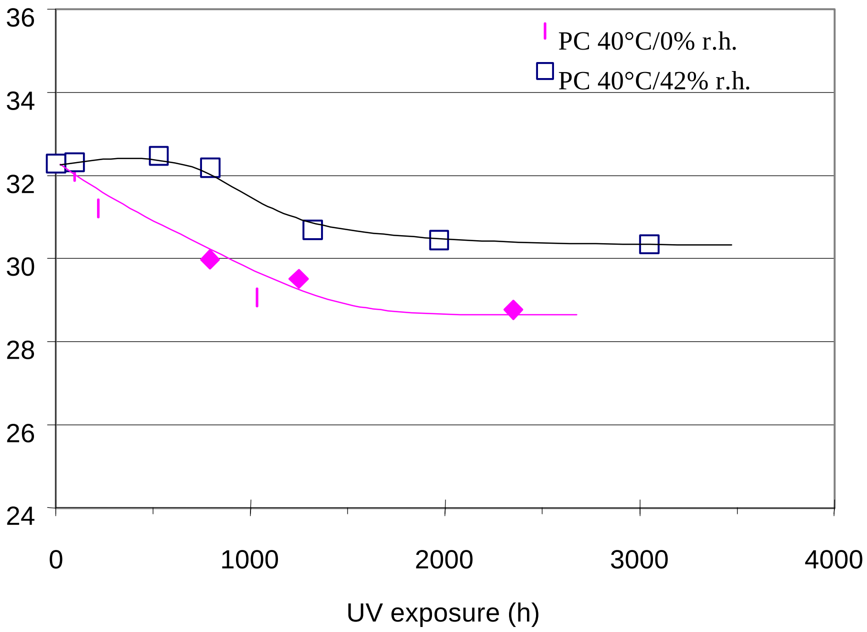


Fig. 4: The effect of UV radiation on molecular weight number and average

3.2 Effect on polymer physical properties

The optical properties were evaluated because of the widespread application of polycarbonate as a glazing material. In such applications uniform light transmission and clarity are fundamental. The light transmission through a material is influenced by factors such as the colour of the sample, the presence of light-scattering bodies in the sample, and the sample surface.

For polycarbonate, the colour of the samples changed upon exposure to UV, initially from a very faint yellow to a stronger yellow, as observed by measuring the change in the yellowness index of the material, as shown in Figure 5. As exposure continued, photo-bleaching occurred, as indicated by the reduction in the value of the yellowness index.

The colour changed progressively and showed a self-accelerating stage, which was characterised by a higher gradient. The curve could be divided into three stages. For samples exposed at relative humidities of 42% and 0%

respectively, the first stage was up to 500 h (42%) and 800 h (0%), the second self-accelerating stage was at 500–800 h (42%) and 800–1400 h (0%), and the third started from 800 h (42%) and 1400 h (0%). Examination of the results for the sample exposed at 42% indicates that it underwent photobleaching after 1500h.

During the first stage, the degradation reactions initiated and Photo-Fries reactions would predominate. In the second stage, the rate of yellowing increased markedly, and in the third stage, the rate of yellowing decreased and a maxim YI value was reached. The changes in the rate of yellowing can be explained by competing degradation reactions. As initial degradation proceeded, the increase in carbonyl groups favoured the absorption of UV. This accelerated the photoreactions and caused the change in slope at stage 2. The photo-oxidation reaction became the predominant reaction after initiation by photo-Fries, then the acceleration of yellowing rate observed in this stage for 42% relative humidity could be explained by Clark and Munro (1983), i.e. that humidity may have assisted in the removal of degradation products from of the polymer, favouring further oxidation.

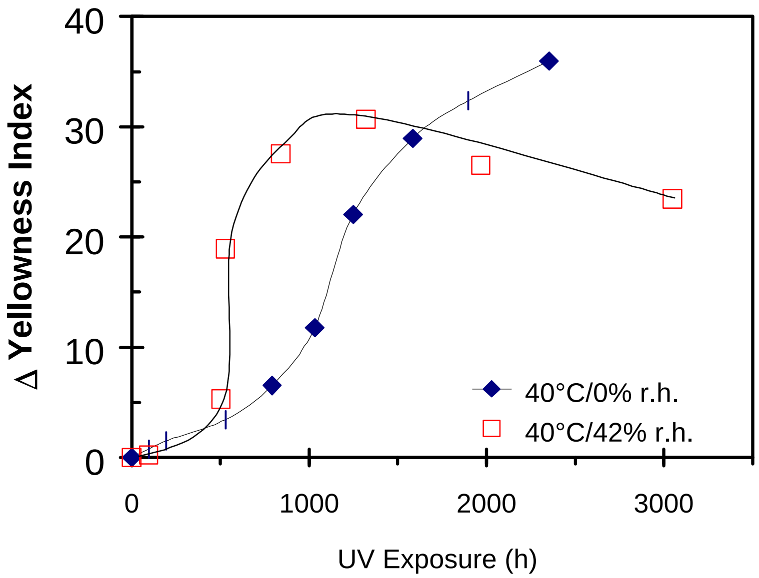


Fig. 5: Effect of UV on the yellowness index of polycarbonate

In the third stage, photobleaching would start to occur, which would mean that the coloured species were being destroyed, i.e. the rate of formation of yellow species was lower than the rate of destruction of the same species. If we assume that the maximum concentration of yellow species is reached, further oxidation of the photoproducts would destroy the conjugated sequences, causing bleaching.

By comparing the results obtained from analysis at low and medium humidity, it was seen that the onset of yellowing was favoured by the increase in relative humidity. Thus, the phenomena reported by Factor and Chu (1980) and Pryde (1985) that humidity prevents degradation by reaction with free radicals was not observed in this work. It is possible that at higher relative humidity levels (>80%) such an effect might be valid, however, this could need to be confirmed.

Our observations do corroborate the results from Rivaton et al. (1986) that humidity accelerates the yellowing process.

At stage 3, a maximum YI was reached for a relative humidity of 42%, whilst for the samples at a relative humidity of 0% the YI kept increasing. Based on the discussion in section 1.2, a possible explanation for this is that the acceleration of photo-oxidation on the outer layers by the higher levels of humidity increased the formation of UV-absorbing species at the polymer surface. This layer then screened the lower wavelength of UV radiation and prevented it from penetrating into the bulk of the polymer and promoting further photoreactions (Factor and Chu 1980). This concept was also discussed by Rivaton (1995), who suggested that as the saturation concentration was reached, the surface layer became fully oxidised. The chromophoric products would then prevent the propagation of the UV radiation into the inner layers of the polymer, thus acting as a barrier against part of the UV range. This would prevent photoreactions in the bulk of the polymer.

For the specimen at a low humidity level, yellowing would occur at a slower rate, allowing the longer wavelengths to penetrate further into the polymer. This would allow a thicker layer of chromophores to be formed, which would eventually allow more chromophores to form than for exposure at high humidities.

As shown in Figure 6, the light transmission of polycarbonate decreased linearly with increased exposure to UV radiation. The reduction in light transmission observed after 2000 h was approximately 4% and 5% for samples aged at 42% and 0% relative humidity, respectively. As the sample turns from transparent to yellow, the polymer absorbs more visible light in the blue range, reducing the total light transmitted through the sample. Therefore, yellowing is partly responsible for the reduction in light transmission. However, other factors such as haze and gloss have to be considered.

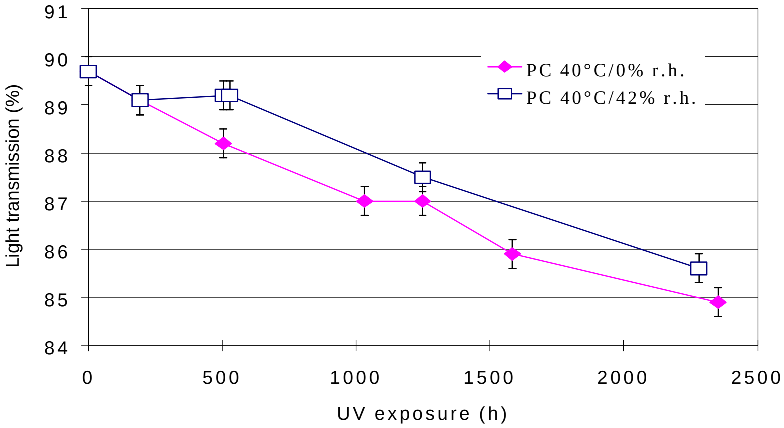
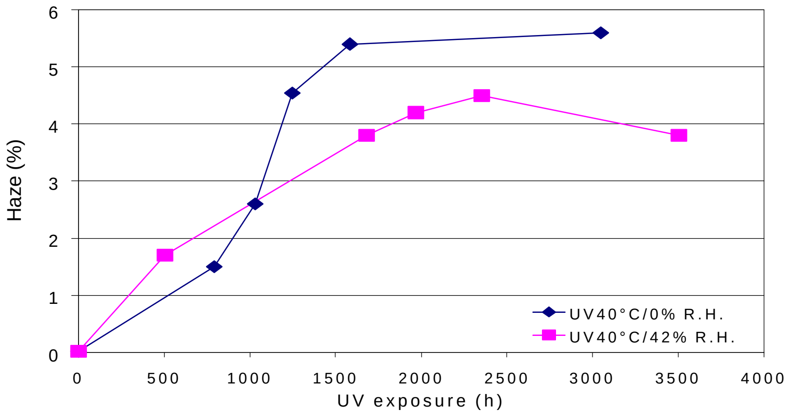


Fig. 6: Effect of UV on the light transmission of polycarbonate

Figure 7 shows that an increase in haze occurs in the samples. The haze increased by slightly less than 6% over a 2000 h period. Haze is a measure of the light scattering of the sample, therefore if the surface becomes uneven or if light - scattering agents are formed in the specimen, the haze values increase and the light transmission values decrease. The samples aged at 0% humidity had a higher haze value than samples aged at 42%, after 1000 h, which would indicate that the phenomenon responsible for haze was more severe at lower humidity.

Fig. 7: Effect of UV on the haze of polycarbonate



As shown in Figure 8, the level of surface gloss of the specimens decreases with exposure to UV, with the reduction being approximately 5% after 2000 h exposure. This would cause more light to be scattered diffusively at the surface of the sample, thus reducing light transmission and increasing haze. The loss in gloss can be explained by examination of SEM micrographs as shown in Figures 9 and 10. It can be seen that the surface of the exposed sample has an eroded appearance, whilst the control sample exhibits a more uniform and smooth surface.

The observations for light transmission, haze and gloss can be related back to our earlier discussion, where at 0% relative humidity the degradation was more severe for all properties measured except yellowing. As discussed above, the reduction in Mw and Mn was larger for samples at 0% relative humidity than for those at 42% relative humidity, indicating that more chain scission and polymer degradation had occurred. As also discussed, the depth of penetration of UV radiation at 0% would be deeper than at 42%, the oxidised layer formed would be thicker and the number of chain scissions per polymer cross-section would be larger. The thicker layer of degraded polymer formed at 0% relative humidity would interfere more strongly with the path of light than the thinner layer formed at 42% relative humidity, giving lower levels of light transmission and increased levels of haze.

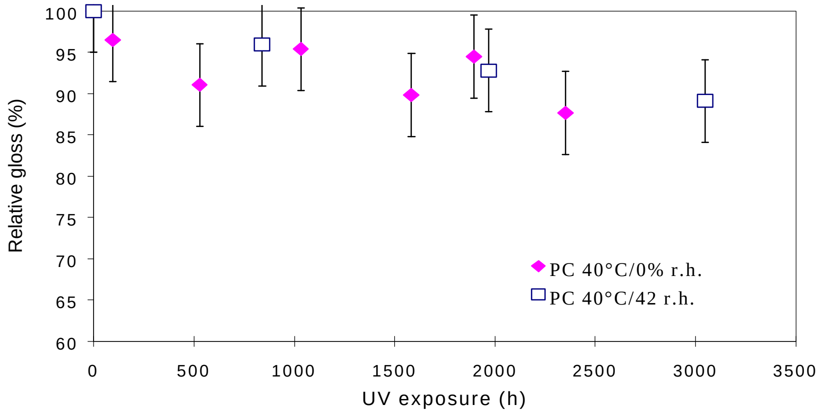


Fig. 8: Effect of UV on the gloss of polycarbonate (relative to the gloss of the control sample)

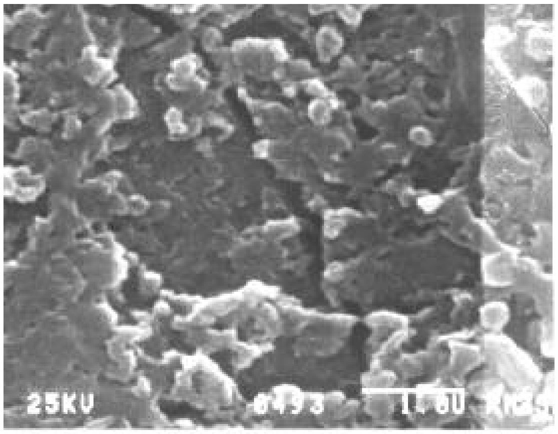


Fig. 9: SEM of polycarbonate exposed to UV for 2350 h

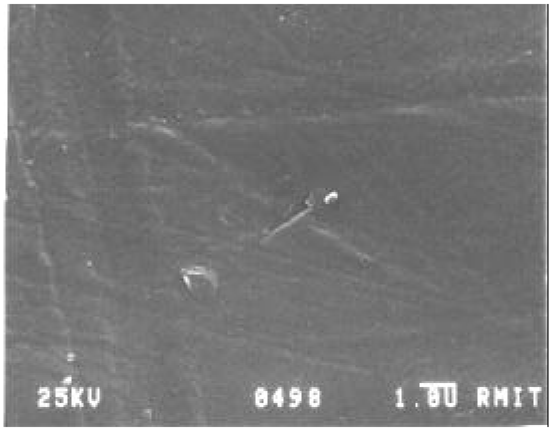


Fig. 10: SEM of polycarbonate control

4 Conclusions

The effect of a range of atmospheric degradation agents on the performance of polycarbonate was investigated in this study. The aim was to determine the effect of these agents, investigate the relationship between physical and chemical changes, and to investigate their degradation mechanisms. The conclusions from our study are:

- Under UVA-340 nm lamps, the mechanism of degradation was identified to be a combination of photo-Fries and photo-oxidation, the later being the predominant reaction over time. This resulted in discoloration, chain scission and consequently a reduction in the molecular weight of the polymer.
- Chain scission at the polymer surface caused embrittlement of the surface, making it more susceptible to erosion. As a result, the polymer surface became uneven, which increased the scattering of light on the polymer surface, reducing gloss and contributing to the increase in haze of the polymer. The increase in

haze and the absorption of visible light due to yellowing decreased the total amount of light transmitted through the polymer.

- Humidity affected the competition between photo-Fries and photo-oxidation, which led to differences in the depth of the degraded layer on polycarbonate. The effect was particularly evident through the changes in yellowing. Yellowing showed that increased humidity affected the rates and mechanisms of the photoreactions.
- At high humidity, yellowing was initially accelerated (the initial YI values obtained were higher than at the low humidity). It then reached a maximum value with time and was followed by photobleaching.
- At low humidity, deterioration in gloss, light transmission, haze, discoloration and molecular weight were more severe. Yellowing increased over time, finally reaching higher values than for the samples at higher humidity.

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