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# Colour Changes of Epoxy Resin Adhesives Used in Glass Restoration by Exposure to $NO_x$ Fumes

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## Abstract

Two-part epoxy adhesive systems (epoxy resin + hardener) are complex mixtures of organic products, which makes identification of the chemical species responsible for discolouration problems very difficult.

Four commercial adhesives currently used by conservators (Araldite®2020, Epo-Tek® 301-2, Fynebond®, and HXTAL®-NYL-1) were investigated. The main confirmations and findings of this work are summarized below.

1. Exposure to $NO_x$ fumes has, in some cases, a significant discolouring effect on the epoxy resin component (part A), pointing to a possible nitration reaction between nitrogen dioxide ($NO_2$) and the aromatic moieties.
2. Exposure to $NO_x$ fumes also has a discolouring effect on the hardener (part B) and in some cases this effect is very strong. This discolouration might be linked to the formation of nitrosamines by reaction of nitric oxide (NO) with secondary amines.
3. Exposure to $NO_x$ fumes causes substantial colouration of cured epoxy adhesives. This factor is probably one of the two main contributors to overall discolouration of epoxy adhesives, together with thermal aging.
4. The epoxy adhesive systems used by conservators are sensitive to moderate heat treatment (80°C for 72 hours), but the discolouration resulting from this treatment is smaller than the one caused by exposure to $NO_x$.
5. Addition of phenolic antioxidant significantly slows down the curing reaction and generally results in some discolouration. This approach does not seem promising to improve the resistance of epoxy adhesives to exposure to $NO_x$ fumes.

## Titre et Résumé

### Altération de la couleur des adhésifs époxydes utilisés en restauration du verre par exposition à des fumées de $NO_x$

Les adhésifs époxydes à deux composants (résine époxyde et durcisseur) constituent des mélanges complexes de produits organiques, ce qui rend très difficile l’identification des composés chimiques responsables des problèmes d’altération de la couleur.

Les quatre produits étudiés sont des adhésifs commerciaux qui sont couramment utilisés par les restaurateurs (Araldite®2020, Epo-Tek® 301-2, Fynebond® et HXTAL®-NYL-1). Un résumé des principaux résultats et des hypothèses confirmées dans le cadre des travaux est présenté ci-après :

1. L’exposition à des fumées de $NO_x$ a, dans certains cas, d’importants effets d’altération de la couleur sur la résine époxyde (composant A de l’adhésif), ce qui pourrait indiquer

une possible réaction de nitration impliquant le dioxyde d'azote ($NO_2$) et les groupements aromatiques;

2. L'exposition à des fumées de $NO_x$ a aussi des effets d'altération de la couleur sur le durcisseur (composant B de l'adhésif), l'altération pouvant, dans certains cas, être très marquée. Celle-ci pourrait être associée à la formation de nitrosamines par le biais d'une réaction de l'oxyde nitrique (NO) et d'amines secondaires;
3. L'exposition à des fumées de $NO_x$ cause une coloration importante des adhésifs époxydes durcis. Ce facteur constitue probablement un des deux principaux éléments qui contribuent à l'altération globale de la couleur des adhésifs époxydes, le second étant le vieillissement thermal;
4. Les types d'adhésifs époxydes utilisés par les restaurateurs sont sensibles au traitement à la chaleur, dans des conditions moyennes (80 °C, pendant 72 heures), mais l'altération de la couleur qu'entraîne ce genre de traitement est moins importante que celle causée par l'exposition à des fumées de $NO_x$;
5. L'ajout d'un antioxydant phénolique ralentit grandement la réaction de durcissement et, de manière générale, entraîne une certaine altération de la couleur. Cette approche ne semble pas très prometteuse comme méthode permettant d'améliorer la résistance des adhésifs époxydes aux effets de l'exposition à des fumées de $NO_x$.

## Introduction

Many polymers show discolouration when aging. The term yellowing is frequently used to describe this phenomenon. However, some polymers yield brownish and sometimes even different colours. We prefer hence to speak of discolouration rather than yellowing.

This discolouration is frequent with epoxy adhesives currently used for glass and non-porous ceramics restoration.

In the case of glass, this phenomenon is particularly unsightly and understanding its origins could contribute to preventive steps to avoid its occurrence.

Epoxy adhesive discolouration can be generated by various oxidation reactions. These reactions are induced by heat and/or light and result in thermal or photo-oxidation.

It is worth noting that epoxy adhesives can exhibit strong discolouration even when totally protected from UV exposure. This topic has been covered by several authors (Bailly 2007; Barlow 1998; Bradley 1990; Down 1984, 1986 and 2001; Shashoua and Ling 1998; Tennent and Townsend 1987). UV-induced reactions will not be discussed in this paper.

These oxidation reactions can affect epoxy resins themselves as well as all other components they contain. Unfortunately understanding these reactions is complicated by:

1. lack of knowledge of the detailed composition of these adhesives coupled with the fact that formulations can be modified by manufacturers without corresponding change in the adhesive's name.
2. appearance of several - sometimes complex- oxidation products; these are difficult to identify as the UV-visible spectra are not really discriminative. Moreover, some of the chemical species produced are characterised by extremely high absorption coefficients which means that colour can appear due to presence of minute amounts of oxidation products.

These two limitations apply indeed in the case of epoxy resins.

Many oxidation agents are active on epoxy resin joins and fills but it is however possible to group them in two well defined categories:

a) Oxygenated free radicals (RO•) and hydroperoxydes (ROOH). These chemical species are generated by chain reactions initiated by free radicals but can also be generated by direct attack of excited oxygen on an organic molecule R-H.

b) Nitrogen oxides NO and $NO_2$ (described under the generic term of $NO_x$). The reactions of these gases with organic molecules are rarely taken into account in previous research, particularly in conservation-restoration literature. These reactions are well known in the textile sector and have even resulted in some experimental procedures to assess their effect which is known as gas fading (AATCC RA33 norm).

The work presented in this paper focuses on the effects of exposure to $NO_x$ fumes and to thermal aging of four epoxy resin adhesives currently used by conservators.

## Choice and Composition of Adhesives

After a survey on the adhesives currently used by Belgian glass conservators, four two-component epoxy adhesives were selected. The chemical composition of these adhesives is given hereunder using the manufacturer's own words (in italics):

1. Araldite 2020. According to the most recent datasheets (dated 28/06/2010) and information communicated by its manufacturer, Huntsman, part A of this adhesive is based on

   a. *butanedioldiglycidyl ether (CAS No 2425-79-8)* at a level between *40* and *52 %*
   b. *reaction product: bisphenol A-(epichlorhydrin); epoxy resin (number average molecular weight < 700) (CAS No 25068-38-6)* at a level between *50* and *62* %)

   Its hardener (part B) is made of

   *c.* *Isophoronediamine (CAS No: 2855-13-2)* at a level between *30* and *60 %*
   *d.* *Trimethylhexamethylenediamine (CAS No: 25620-58-0)* at a level between *13* and *30 %*
   e. A third undisclosed component used as a viscosity adjuster and terminated by a secondary amine. We suspect that this component could be an amine terminated butadiene-acrylonitrile copolymer (ATBN) (Petrie 2006, p. 147-149) terminated for instance by N-aminoethylpiperazine (CAS No: 140-31-8)

   According to its producer, Araldite 2020 does not contain phenolic antioxidant.

2. Epo-Tek 301-2. According to the most recent datasheet from its manufacturer, Epoxy Technology, Inc., (dated 12/05/2008), part A of this adhesive is based on *diglycidyl ether of bisphenol A (CAS No 1675-54-3)* at a level of *75-100%*.
   Its hardener (part B) is made of *polyoxypropylenediamine (CAS No 9046-10-0)* at a level of *75-100 %*.

3. Fynebond. According to the most recent datasheet from its manufacturer, Fyne Conservation Services (FCS), part A of this adhesive is made of *liquid epoxy resin of the*

*bisphenol A, epichlorohydrin type (Mn <= 700), specifically bis (4-(2,3-epoxpropoxy) phenyl ) propane*.
Its hardener (part B) is a *difunctional primary amine; a polyoxypropylenediamine whose amine groups are located on secondary carbon atoms at the ends of the aliphatic polyether chain*.

4. HXTAL NYL-1
   According to the most recent datasheet from its manufacturer, HXTAL Adhesive, LLC, (dated 11/10/2010), part A of this adhesive is made of *100 % of epoxy resin (CAS No 30583-72-3)* (i.e. a reaction product of perhydrogenated bisphenol A with epichlorohydrin) with traces of *diglycidyl ether ( CAS No 2238-07-5)* and *1-chloreo-2,3-epoxypropane (CAS No 106-89-8)*.
   Its hardener (part B) is made of
   a. *poly(oxy)(methyl-1,2-ethanediyl), alpha-hydro-omega-(2-aminomethylathoxy)-ether 2-ethyl-2-(hydroxymethyl)-1,3-propanediol (3 :1) – (CAS No : 39423-51-3)* (for a graphical representation of this polyetheramine containing repeating oxypropylene units see for instance *Technical bulletin* from Huntsman 2007) at a level of *< 92 %*.
   b. *1,3-diaza-2,4-cyclopentadiene, glyoxaline (CAS No 288-32-4)* (i.e. imidazole a heterocyclic molecule in which the nitrogen atom in position 3 is a secondary amine) at a level of *< 10 %*.
   According to its producer, HXTAL NYL-1 does not contain phenolic antioxidant.

The composition of these adhesives is summarised in Table 1:

Table 1: Composition of the four adhesives tested

| Adhesive | Epoxy Resin (part A) | Hardener (part B) |
|---|---|---|
| Araldite 2020 | Partly aromatic - partly aliphatic | Aliphatic (low molecular weight) diamines |
| Epo-Tek 301-2 | Predominantly aromatic | Polyether diamine |
| Fynebond | Aromatic | Polyether diamine |
| HXTAL-NYL-1 | Aliphatic | Polyether triamine + heterocyclic amine |

It should be noted that:

a. composition of every epoxy resin adhesive is complex; moreover industrial organic compounds, particularly polymeric ones, are practically never pure single substances;
b. complete adhesive compositions are not known at least for the two first adhesives listed above. It may be suspected that the missing component(s) could also contribute to the adhesive’s discolouration.

The curing mechanism of these four epoxy resin adhesives should be similar and is described in any textbook on epoxy adhesives (Petrie 2006, p. 37-38). It implies when starting from either a primary amine or a secondary amine a first reaction with an epoxy group giving chain extension. This is followed in the case of primary amines by a second reaction with another epoxy group leading to cross-linking (Figure 1):

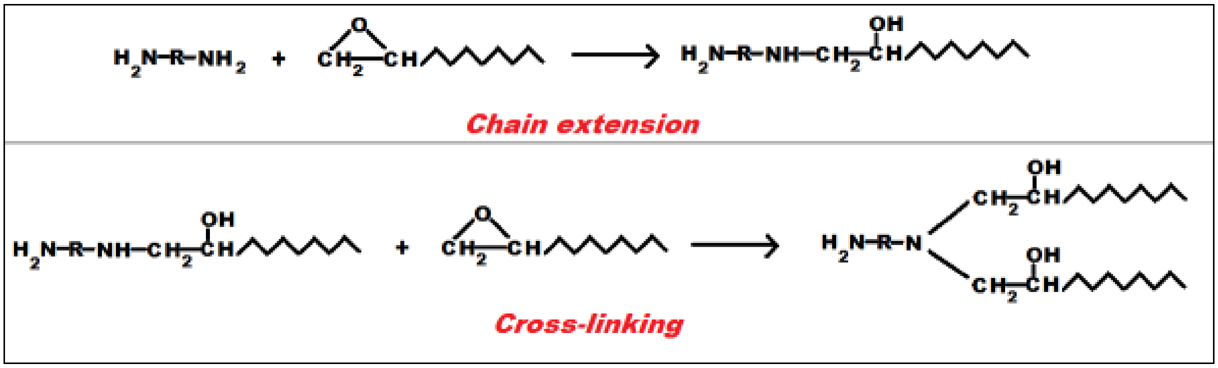

*Figure 1: Chain extension and cross-linking mechanisms for epoxy resin adhesives*

# Experimental

## Sample preparation

Test specimens (20 x 50 x 3 mm) were prepared by casting in a mould a mixture of part A and part B of each adhesive using mixing ratios recommended by the manufacturers.

Samples of the four epoxy resin adhesives were also prepared with a phenolic antioxidant, Lowinox 1790. This additive was added to the hardener in an amount that would make it 1% after mixing with the epoxy resin. Lowinox 1790 is known to be very effective against free radicals and also to be used as a $NO_x$ scavenger in some polymers (Lowinox 1790 Technical Data Sheet 2008).

## Exposure to $NO_x$ fumes

Exposure to $NO_x$ fumes was carried out by placing the tests specimens and/or open pots containing either part A or part B of the adhesives selected on a desiccator plate. A beaker containing 2 g of finely powdered copper was placed at the centre of this desiccator plate.

$NO_x$ production was initiated by pouring 5 ml of nitric acid at 65 % on the powder (see Figure 2). This operation must be carried out carefully as the reaction is very violent. The desiccator was then rapidly shut down and kept closed during two hours allowing $NO_x$ to react with the samples to be tested. After this time the desiccator was open and the remaining $NO_x$ fumes evacuated.

The amount of $NO_x$ produced is about the same for each exposure experiment and seems to have been nearly totally consumed after two hours. Consequently, the degree of discolouration is proportional to the number of tests samples put in the desiccator. This number will be indicated in the results section.

*Figure 2: Production of $NO_x$ by reaction of copper powder with nitric acid.*
*Left: pouring of nitric acid in the beaker; right: $NO_x$ fumes in the desiccator*

## Oven aging

A set of test specimens made with the four selected epoxy resin adhesives was aged in a dry oven at 80°C for 72 hours. This temperature and time were selected to achieve a complete polymerisation of the adhesives.

## Colour measurements

Colour of the samples treated could be easily assessed by visual inspection. However to try and be more quantitative, and also to try and avoid the problems caused by lighting when taking photos, the following procedure was developed:

Each of the test specimens to be measured was placed always in the same position on a white background under three 500 Watt spot lights. Photos were taken with the camera settings fixed manually on the same values. The photos were examined using Paint.NET "*Colors Window - More Mode*" option. This tool allows measuring the RGB (Red, Green, Blue) values on any selected point. Twelve points were measured on the photo of each test specimen and the average was calculated. This gives a measure of R, G or B on a scale having a maximum value of 255 for each colour. The three components of a RGB triplet are encoded as integer numbers in the range 0 to 255, this gives 256 distinct values. In this scale, pure white is characterised by R = G = B = 255. Any colour development will be measured by a decrease of the value of either R, G or B.

These measurements were carried out on test specimens untreated and treated by exposure to $NO_x$ fumes or oven aging. The differences $\Delta R$, $\Delta G$ and $\Delta B$ were measured. The Global Colour Change was defined as $(\Delta R + \Delta G + \Delta B)$.

# Results

## a) Exposure to $NO_x$ Fumes of Part A (Epoxy Resins) of the four Adhesives

Exposure to $NO_x$ of part A (containing the epoxy resin components) of each of the four adhesives gave the following results (Figure 3):

*Figure 3: Part A epoxy resin before (upper row) and after (lower row) exposure to $NO_x$. From left to right: Araldite 2020, Epo-Tek 301-2, Fynebond and HXTAL NYL-1.*

The discolouration assessed visually (see Figure 3) increased in the following sequence:

HXTAL NYL-1 < Araldite 2020 < Epo-Tek 301-2 < Fynebond

The reactions involved here are probably nitration of the aromatic moieties by $NO_2$ (Albright 2000). It is therefore normal that an aliphatic (i.e. not containing aromatic moieties) epoxy resin like the one used in HXTAL NYL-1 and a partly aliphatic resin like the one used in Araldite 2020 will show less discolouration than predominantly aromatic resins like those used in Epo-Tek 301-2 and Fynebond.

## b) Exposure to $NO_x$ Fumes of Part B (Hardeners) of the four Adhesives

Exposure to $NO_x$ of part B (hardener) of each of the four adhesives gave the following results:

*Figure 4: Part B (hardeners) before (upper row) and after (lower row) exposure to $NO_x$.*
*From left to right: Araldite 2020, Epo-Tek 301-2, Fynebond and HXTAL NYL-1.*

Discolouration assessed visually (see Figure 4) was clearly observed in all hardeners and was particularly strong in the case of Araldite 2020.

A possible explanation for this phenomenon could be the following: part B contains amine compounds. Secondary amines are known to react with NO to give highly coloured nitrosamines according to the following reaction (Challis and Butler 1968):

$$R_1R_2\text{-NH} + \bullet NO \rightarrow R_1R_2N\text{-}N{=}O + H\bullet$$

Therefore, if secondary amines were present in part B, colour will develop. Those secondary amines can be present as such in the hardener or as impurities arising from the amine production route (Turcotte and Hayes 2001).

This discolouration however is not really relevant to the aging of epoxy adhesives as the amines contained in the hardener will react with the epoxy resin (part A) during the curing reaction. Nevertheless it does show very clearly why it is so important to use the right proportions when blending these two-component epoxies. Excess hardener can significantly contribute to a $NO_x$-induced discolouration process.

### c) Exposure to $NO_x$ Fumes of the Cured Test Specimens made with the four Adhesives

This experiment is probably the most relevant to assess the effect of $NO_x$ pollutants on epoxy adhesives. In this experiment ten test specimens were put in the desiccator: one for each resin tested and six other test specimens for another experiment not reported in this paper.

The results observed are presented in Figure 5:

*Figure 5: Epoxy adhesives test specimens before (upper row) and after (lower row) exposure to $NO_x$. From left to right: Araldite 2020, Epo-Tek 301-2, Fynebond and HXTAL NYL-1.*

In Figure 5, it is clearly seen visually that some adhesives show stronger discolouration than others. The global colour change, which is the sum of all the changes in red (ΔR), green (ΔG) and blue (ΔB) components, for all four adhesives is shown in Table 2 and Figure 6.

Table 2: Colour change resulting from exposure to $NO_x$ measured by RGB measurements

| Adhesive | ΔR | ΔG | ΔB | ΔGlobal |
|---|---|---|---|---|
| Araldite 2020 | -11 | -10 | -63 | -84 |
| Epo-Tek 301-2 | -7 | -18 | -91 | -116 |
| Fynebond | -14 | -37 | -111 | -162 |
| HXTAL-NYL-1 | -8 | -8 | -24 | -40 |

It is clearly seen that some adhesives show stronger discolouration than others. Quantitatively this could be plotted in a graph as follows (Figure 6):

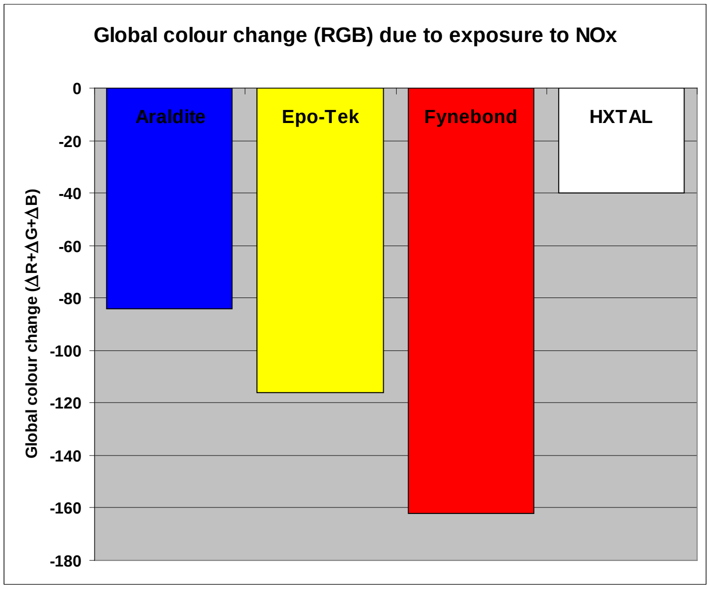

*Figure 6: Global colour change*
*i.e. sum of the decreases in R, G and B values measured on test specimens shown in Figure 5.*

The colours appearing after a short exposure to $NO_x$ fumes are indeed quite strong. The adhesive showing the least colour change in this test is HXTAL-NYL-1; it is followed by Araldite 2020.

The following hypothesis can be made to explain these colour changes which may be due to the addition of two processes:

a) nitration reaction of aromatic moieties with $NO_2$ as already discussed for part A exposure experiment
b) formation of nitrosamines by reaction of NO with secondary amines. Indeed, if after curing, secondary amines are still present in the adhesive, they are likely to react with NO giving strongly coloured nitrosamines. These secondary amines can be generated from insufficient cross-linking reaction or from excess hardener.

As $NO_x$ is a common atmospheric pollutant, this could be one of the main causes of epoxy resin adhesives discolouration under natural aging conditions. Of course it should be noted that the amounts of $NO_x$ coming from atmospheric pollution (typically under 1 ppm) are very small compared to the massive exposure achieved in this experiment.

## d) Effect of Oven Aging

Oven aging was carried out at 80°C for 72 hours and as described in the literature (Barlow 1998; Bradley 1990; Down 1984, 1986 and 2001; Shashoua and Ling 1998), this mild aging produced noticeable discolouration to our samples (Figure 7):

*Figure 7: Test specimens before (upper row) and after (lower row) oven aging*
*From left to right: Araldite 2020, Epo-Tek 301-2, Fynebond and HXTAL NYL-1.*

All adhesives were affected with slightly less discolouration for HXTAL NYL-1 and Fynebond.

The colour changes in this experiment, assessed by visual examination and measured by global colour change ($\Delta R + \Delta G + \Delta B$), are moderate compared to the changes achieved in the $NO_x$ exposure experiment. This is shown in Table 3 and in Figure 8:

Table 3: Comparison measured by RGB measurements of colour change resulting from oven aging (80°C, 72 hours) with colour change caused by exposure to NOx

| | Oven Aging | | | | $NO_x$ exposure | | | |
|---|---|---|---|---|---|---|---|---|
| Adhesive | ΔR | ΔG | ΔB | ΔGlobal | ΔR | ΔG | ΔB | ΔGlobal |
| Araldite 2020 | -6 | -7 | -10 | -23 | -11 | -10 | -63 | -84 |
| Epo-Tek 301-2 | -5 | -6 | -13 | -24 | -7 | -18 | -91 | -116 |
| Fynebond | -4 | -4 | -12 | -20 | -14 | -37 | -111 | -162 |
| HXTAL-NYL-1 | -1 | -3 | -13 | -17 | -8 | -8 | -24 | -40 |

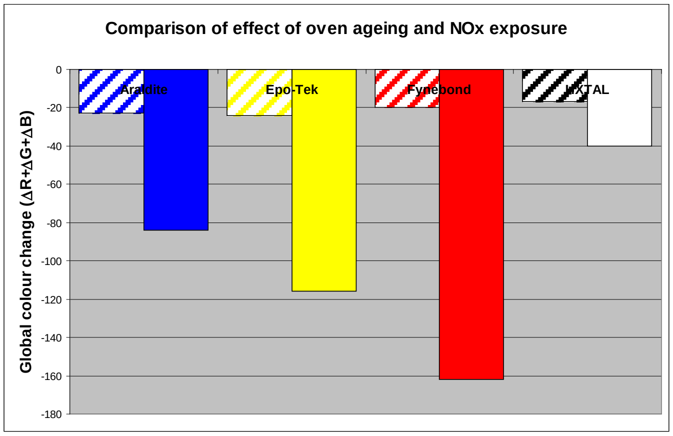

*Figure 8: Comparison of colour changes of test specimens as measured by (ΔR + ΔG + ΔB) Hatched colour = effect of oven aging. Plain colour = effect of $NO_x$ exposure.*

This difference shows indeed that exposure to $NO_x$ can in some cases have a stronger effect than pure thermal aging. Of course, the relativity of this statement should be kept in mind. The conditions used are far away from storage conditions in museums and the tests were only carried out with a massive exposure to $NO_x$ for two hours and an oven aging at a single temperature (80°C) and time (72 hours). Ideally to understand the relative effect of atmospheric $NO_x$ exposure and thermal aging different concentrations of $NO_x$ should be tested as well as several temperatures and/or times for oven aging.

## e) Reaction with $NO_x$ of the Test Specimens made with the four Adhesives Stabilised with 1% of Antioxidant Lowinox 1790

To try and prevent discolouration induced by NOx we have added to the adhesives large quantities (1%) of an antioxidant, Lowinox 1790, known to react strongly with $NO_x$ but without giving coloured reaction products.

Addition of Lowinox1790 to the hardeners resulted in a very strong slowing down of the curing process. This may be an indication of inhibition of a polymerisation reaction involving free radicals.

This slowing down was so high that it prevented the production of completely cured test specimens made with Epo-Tek 301-2 and Fynebond. On the other hand the addition of such a large amount of phenolic antioxidant gave already - by itself and before submitting the samples to $NO_x$ - a discolouration of either the hardeners alone (part B) or of the cured test specimens. This discolouration was quite accentuated by exposure to $NO_x$ fumes.

These results show that the addition of an antioxidant specifically aimed at scavenging $NO_x$ has a strong effect on the curing rate and an adverse effect on the colour development. Consequently this approach does not seem promising to improve the resistance to discolouration of epoxy adhesives.

## Conclusions

Exposure to $NO_x$ fumes is definitely a factor contributing to two-part epoxy adhesives discolouration. $NO_x$ being a universal air pollutant this discolouration is likely to affect unsightly transparent substrates like glass that are adhered with these types of adhesives. This can be a real problem as these adhesives are commonly used in glass restoration either to bond together parts or to make filings.

Our understanding of the chemical reactions implied is complicated by the complex composition of these adhesives and by the fact that some components are not disclosed by manufacturers.

At this stage, we can make the hypothesis that two mechanisms are probably responsible for this discolouration:

1. nitration by $NO_2$ of the aromatic moieties of epoxy resin contained in part A,
2. reaction of NO with secondary amines still present in the polymer after curing.

Knowing the main requirements for an ideal epoxy adhesive for conservation, it could be worthwhile to try and formulate it taking into account the purity of the various components used and their susceptibility to $NO_x$.

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# Materials and Suppliers

**Epoxy resin adhesives**

- Araldite 2020 is manufactured by Huntsman International, LLC. For addresses of distributors see: http://www.huntsman.com/advanced_materials/eng/Customer_Service/Global_customer_service/index.cfm?PageID=5785, accessed May 23, 2011.
- Epo-Tek 301-2 is manufactured by Epoxy Technology, Inc. For addresses of distributors see: http://www.epotek.com/find-rep.asp, accessed May 23, 2011.
- Fynebond is manufactured by Fyne Conservation Services - Airds Cottage, St. Catherine', Loch Fyne, Argyll, Scotland PA25 8BA U.K. Contact: http://www.fyne-conservation.com/component/option,com_contact/Itemid,3/, accessed May 23, 2011.
- HXTAL-NYL-1 is manufactured by HXTAL Adhesive, LLC for addresses of distributors see http://www.hxtal.com/distributors/distributors.html, accessed May 23, 2011.

**Phenolic antioxidant**

Lowinox 1790 is 1,3,5-tris(4-tert-butyl-3-hydroxy-2,6 dimethylbenzyl)-1,3,5-triazine-2,4,6-(1H,3H,5H)-trione (CAS N°40601-76-1). It is manufactured by Chemtura. Contact: "Antioxidant and UV Stabilizer Solutions" tel: +41 52 723 4417 e-mail: Sergio.Palumbo@chemtura.com, (accessed May 23, 2011).

**Paint.NET software**

This free image and photo editing software can be downloaded from http://www.getpaint.net/, (accessed May 23, 2011). According to its website "*It started development as an undergraduate college senior design project mentored by Microsoft, and is currently being maintained by some of the alumni that originally worked on it. Originally intended as a free replacement for the Microsoft Paint software that comes with Windows, it has grown into a powerful yet simple image and photo editor tool.* "

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