

## **CONTROLLING THE REFRACTIVE INDEX OF EPOXY ADHESIVES WITH ACCEPTABLE YELLOWING AFTER AGING**

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**ABSTRACT**—Mixtures of Ablebond and HXTAL NYL-1 were found to produce epoxies with a range of refractive indexes from 1.515 to 1.565. Their yellowing after dark aging is measured and determined to be acceptable for glass repair.

### **1 INTRODUCTION**

In repairing a broken glass object, factors that will make the repair less visible are usually considered. One such factor, important in the repair of colorless glass, is the approximate match between the index of refraction of the adhesive and that of the glass (Ogden 1975; Tennent and Townsend 1984a; Messinger and Lansbury 1989). Tennent and Townsend (1984b) describe a glass repair where the difference between the index of refraction of the glass and that of the adhesive is 0.04; this repair is clearly visible. However, in another repair of the same glass, the difference of refractive indexes is approximately 0.01, and the repair is not easily visible under normal lighting conditions.

Recently, Messinger and Lansbury (1989) developed a method of controlling the index of refraction of epoxy adhesives to a small degree. This control was achieved by adding a mixture of 1,2-epoxy-3-[2,4,6-tribromophenoxy] propane and 1,2-epoxy-3-[2,4,6-triiodophenoxy] propane to either of two commercially available epoxies. Ablebond mixed with such additives produced a range of refractive index from 1.565 to 1.59. The resulting adhesives were found to have yellowing characteristics within acceptable standards when aged at room temperature in the dark as defined by Down (1984). HXTAL NYL-1 mixed with such additives produced a range of refractive indexes from 1.515 to 1.545. However, the halogenated additives caused the adhesives made with HXTAL NYL-1 to yellow beyond the acceptable standards.

This study was conducted to determine whether mixtures of Ablebond resin and HXTAL NYL-1 resin could be used to produce epoxies with a range of refractive index from 1.515 to 1.565, which also pass the standards of Down (1984) for yellowing.

### **2 EXPERIMENTAL**

HXTAL NYL-1 resin and Ablebond resin were mixed in a variety of weight percent (100:0, 87.5:12.5, 75.0:25.0, 62.5:37.5, 50.0:50.0, 37.5:62.5, 25.0:75.0, 12.5:87.5, 0:100). Two separate sets of mixtures were prepared, from two separate resin batches.

Each resin mixture was prepared in a clean, glass test tube and stirred thoroughly with a glass rod. The HXTAL NYL-1 catalyst was added to each total resin mixture, in a proportion of 4:1 (resin:catalyst) by weight. Again, each resin mixture was stirred thoroughly with a glass rod. Preliminary tests, not reported here, indicated that if either of the two stirring procedures (blending the resins and adding the catalyst) were not thorough, then the resulting cast epoxies did not have uniform hardening or yellowing characteristics.

All epoxy mixtures were cast into small blocks measuring 3 × 12 × 20 mm. This size is required to fit the sample window on the Abbe refractometer. These mixtures were cured at 59–60 °C for 6 hours.

After cooling overnight, the indexes of refraction were measured with an Abbe refractometer, according to the method of Messinger and Lansbury (1989). Refractive indexes were similarly measured after one year of dark aging. The UV-visible absorbance of the adhesive blocks was also measured after one year of dark aging, according to the method of Messinger and Lansbury (1989). As in their study, the degree of yellowing was represented by the value of  $A_t$ , calculated according to the method of Down (1984).<sup>1</sup>

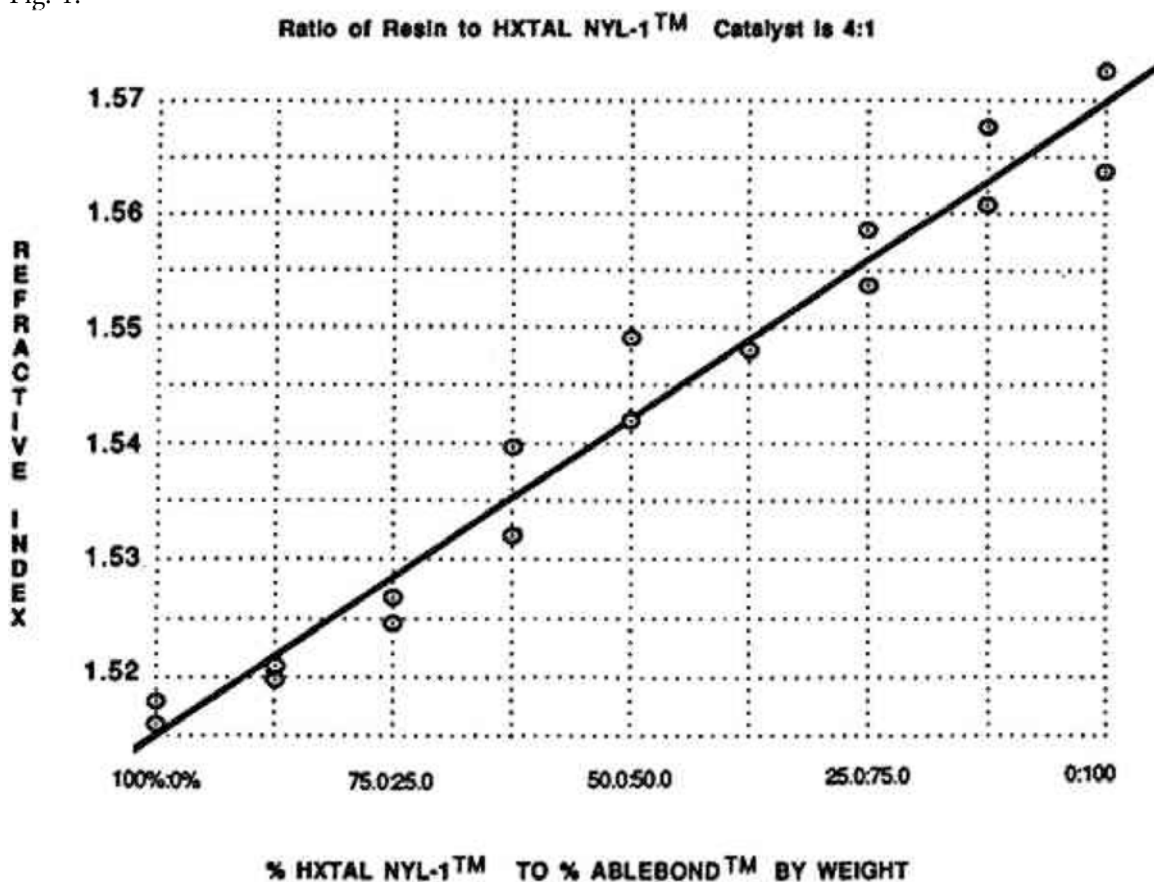
### 3 RESULTS

All samples prepared were found to harden into rigid solid blocks as expected (manual flexing could not crack the blocks and a fingernail could not dent them).

#### 3.1 INDEXES OF REFRACTION

Figure 1 illustrates the data for the initial measurements of refractive indexes of the different adhesive mixtures (prior to the aging period).

Fig. 1.



#### 3.2 CHARACTERISTICS AFTER ONE YEAR OF DARK AGING

After aging, no change in the refractive index could be observed beyond the limits of accuracy of the Abbe refractometer.

At values calculated from the UV-visible absorbance of the adhesives after aging were, in all cases, acceptable according to the criteria set forth by Down (1984) ( $A_t > 0.1$ ). In general, adhesives tended

to become more yellow with increased concentration of Ablebond resin. For the mixtures containing only 12.5% Ablebond resin (87.5% HXTAL NYL-1 resin), the yellowing was quite negligible, with  $A_t = 0.0063$ . The single most yellowed sample, which was one of those containing 100% Ablebond resin, was still quite acceptable, with  $A_t = 0.0277$ .

#### 4 CONCLUSIONS

It is possible to adjust the index of refraction of epoxy adhesives within the range 1.515–1.57 by varying the ratio of amounts of Ablebond resin to HXTAL NYL-1 resin. When the adhesives were subject to one year aging in the dark at room temperature, the amount of yellowing of the cured adhesives was found to be within acceptable limits as established by Down (1984). Many other properties of these modified epoxies, including their physical strengths, remain untested. The authors encourage the study of these and similarly modified epoxies to develop a clear understanding of their suitable and unsuitable applications in glass conservation. Any recommendation of their use, except in experimental situations, should await these tests.

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#### NOTES

. According to Down (1984) method, the degree of yellowing is represented by the value of  $A_t$ , as calculated by the following equation:

$$A_t = (A_{380 \text{ nm}} - A_{600 \text{ nm}}) (0.1 \text{ mm}/F)$$

where  $A_t$  = the degree of yellowing after time t

$A_{380 \text{ nm}}$  = the absorbance at 380 nm  
after time t

$A_{600 \text{ nm}}$  = the absorbance at 600 nm  
after time t

F = the thickness of the sample in mm

Fig. .

Samples with  $A_t$  values of less than 0.1 were always considered “acceptable” in color (as determined by a group of observers).  $A_t$  values between 0.1 and 0.24 were considered to be of “questionable acceptability.”  $A_t$  values greater than 0.25 were always considered to be “unacceptable” in color.

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