ARALDITE—A LABORATORY ASSESSMENT

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(*Received* 23 *April* 1969; *revised* 12 *August* 1969)

The suitability of Araldite, an indigenous thermosetting epoxy resin, as an optical cement in place of Canada Balsam and also as a general purpose adhesive for bonding glass to metal joints in optical instruments, has been determined under laboratory conditions.

Araldite is a thermosetting epoxy resin marketed by M/s CIBA of India Ltd. It is available both as a viscous liquid and as paste. The liquid is almost colourless and the paste has a light bluish tinge. The hardener used with araldite is also supplied either in liquid form or in paste and is light yellowish-green.

Araldite and the hardener are mixed in definite proportions (e.g. 100 parts of Araldite AY103 and 8 to 9 parts of hardener HY951 by weight). The mixture has been tried as an optical cement and also as a general purpose adhesive for bonding glass to metal joints in optical instruments. The present report describes the behaviour of the material under laboratory conditions. The report is given in two parts : Part I—Araldite as an optical cement and Part II—Araldite as a general purpose adhesive.

ARALDITE AS AN OPTICAL CEMENT

Qualitative Requirements[1, 2]

An optical cement should broadly fulfil the qualitative requirements mentioned in Appendix 1.

Test Pieces

A number of lens doublets (approx. 2·5 cm. dia.) and glass strips (microscope slides) of assorted sizes were used as test pieces for trials.

The liquid Araldite AY103 was mixed with the liquid Hardener HY951 in the proportion 100 : 8 to 9 parts by weight and immediately used to cement the test pieces. After cementing, the test pieces were cured for one hour at 100°C in an electric oven. Appendix 2 gives the various conditions under which curing can be done.

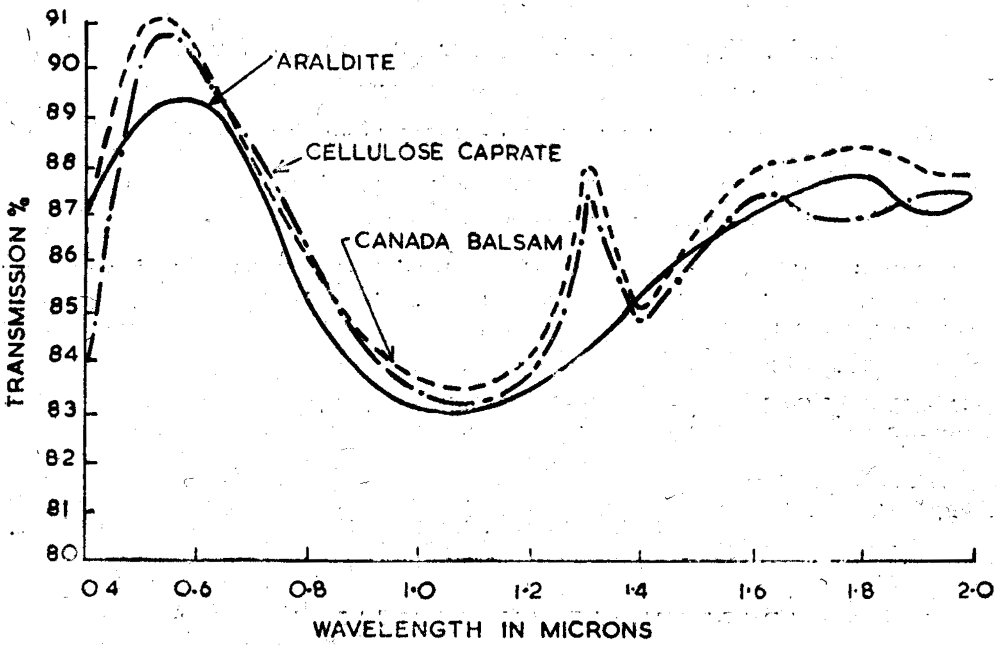
A few small blocks/discs, (rectangular and circular) were also made of the material, cured and afterwards polished in an optical polishing machine.

A small quantity of the mixture was also kept in a watch glass covered with a glass plate at room temperature (22°C) to check its pot-life or shelf life.

Tests and Results

Test pieces were subjected to the various tests mentioned below and observations made. The climatic tests were largely based on the Indian Standard Specification No. IS : 2352/1963.

Colour : A visual examination was made of the polished blocks of different thicknesses and cemented doublets. The blocks appeared light yellow in colour. No colour was noticed in the cemented doublets.



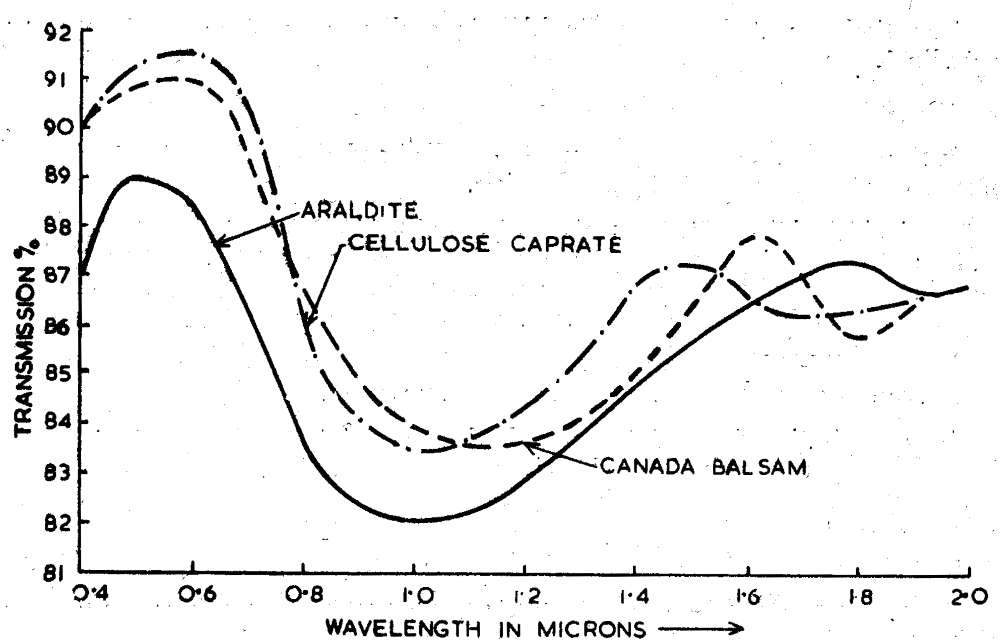


Fig. 1—Transmission characteristics of Araldite *vs* Cellulose Caprate and Canada Balsam for two samples.

Transmission: Araldite discs of 2 mm. thickness (when polished) were made and transmission in white light was measured with a Baldwin photometer. 81% transmission (average of 5 readings) was observed.

Two doublets, one cemented with Canada Balsam and the other with Araldite were tested by Baldwin photometer. 90% transmission was observed in both the doublets.

Microscope slides of 1 mm. thick each were cemented and transmission characteristics measured in the range of 0·4–2·0 microns by spectrophotometer. The characteristic curves of Araldite in comparison with Canada Balsam and Cellulose Caprate are shown in Fig 1. Transmission is found to be very low in the infra-red region.

Refractive index: Abbe's refractometer was used to determine refractive index which was found to be 1·5798 (average of 15 readings) at 31°C dispersion $n_f - n_c = 0{\cdot}17376$ (average of 15 readings).

Fluorescence and phosphorescence: Cemented doublets and polished blocks were exposed to ultra-violet light in a dark room. Cemented blocks did but doublets did not exhibit fluorescence. No phosphorescence was observed in any case.

Homogeneity, strain and shrinkage: Polished discs of 2-4 mm. thickness were viewed through microscope and finished doublets through strain viewer. They were observed to be homogeneous. No strain or shrinkage was noticed.

Resistance to extreme cold/dry, heat/thermal shock: The cemented doublets were subjected to (i) extreme cold at —40°C for 4 hours, (ii) dry heat at 75°C for 16 hours in an electric oven, and (iii) extreme cold (—40°C) and dry heat (75°C) alternatively for 3 hours each. No deterioration was observed in either case.

Resistance to moisture, UV radiation and damp heat: The doublets were (i) immersed in water for several days, (ii) exposed to ultraviolet light for 100 hours and (iii) exposed to 55°C and 95-100% RH for 24 hours. This cycle was repeated 20 times. No deterioration was observed in either case.

Resistance to fungus growth: Araldite (paste and liquid) was smeared on petri dishes, cured and inoculated with fungal spores. Dishes were kept in humid atmosphere for one week. Empty dishes as control samples were kept separately under similar conditions. No fungus growth was observed on Araldite although the control samples showed profuse growth.

Ageing: Doublets were kept in a room for approximately one year. No deterioration or chemical instability was observed.

Adhesive strength: Cemented doublets were dropped from a height of 180 cm. onto a cemented floor for a number of times. Adhesion was found to be satisfactory as joints were not affected though glass was found cracked/chipped off.

Chemical inertness: The cement was put on different materials such as plastic, rubber hylam, copper, brass, aluminium, iron and glass. No deterioration was observed at the contact surfaces.

Applicability: The cement was prepared and surfaces bonded and cured as per instructions contained in Appendix 2. The cement was easy to apply. Once cured, truing could not be done. Careful jigging was essential while curing.

Decementing: The jointed doublets did not decement when heated at 200°C for 6 hours or immersed for several days in nitro-benzol, trichloroethylene, acetone, cresol. phenol, petrol, spirit, benzene, xylene, conc. HCl, H_2SO_4 and HNO_3.

The method of separation by using epoxy resin remover (for details see Appendix 3) did not give consistent results in the sense that the time of separation was varying. Even otherwise, the use of conc. HNO_3 was not desirable as it affects glass.

Under advice from M/s CIBA of India Ltd., the hardener HY930 was used in place of HY951 and cemented doublets were heated to 200°C for several hours. One doublet separated after 6 hours. Two others did not disengage even after 16 hours. Brown lines appeared in one of these showing charring.

Storage life: A small quantity of mixture, when kept on watch glass under ambient conditions, hardened after 1½ hours showing that storage life was very limited. However, the raw materials—Araldite and hardener—though stored for 18 months did not show any sign of deterioration.

ARALDITE AS A GENERAL-PURPOSE ADHESIVE

The bonding property of Araldite is well-known[3,4] and has also been established here under "Araldite as an Opticle Cement". Twyman[5] has described Araldite as a good synthetic resin adhesive for bonding metals, glass and other materials. However, a report came to our notice that sealed optical instruments using Araldite to join glass to metal parts, showed fogging when the instruments were tested under dry heat conditions. The hygroscopic property, if any, of Araldite was, therefore, investigated by conducting the following tests:

Tests and Results

Two tests were carried out as detailed below and observations made:

Test No. 1: Small quantities of Araldite with hardener in specified proportions were weighed in watch glasses and cured at 100°C for 1-3 hours, weighed when cooled, and then left in ambient conditions for a period of 16-40 hours and re-weighed. They were again heated for 1 hour at 100°C, weighed when cooled and kept in ambient conditions for 16-40 hours and re-weighed. This cycle was repeated several times. A loss in weight was observed after every heating and a gain in weight when left in ambient conditions. The loss or gain was 0·15 to 0·25 per cent.

Test No. 2: Observations were also made with small brass tubes (2-5 cm. in dia. and 6-10 cm. in length), smeared with a small portion of the cement on their inner walls and sealed at both ends with glass plates using Araldite mixture. The cement was cured before the tubes were sealed and observations made after keeping the sealed tubes in an electric oven at 100°C for 1 hour and also at 70°C for several hours. Empty sealed tubes were used as control samples.

Fogging was observed on the glass plates when the tubes were heated and taken out of the oven. No fogging was observed on the controls showing that fogging was not due to any other extraneous matter.

(Note :—All tubes and sealing glass plates were dried by heating before use. Both liquid and paste Araldite were used in the experiments).

DISCUSSION

From the results given above, it may be stated that Araldite AY103 (mixed with hardener HY951) is a good optical cement, but that it stands at a disadvantage in comparison with the traditional cement, Canada Balsam, from the point of view of decementing. Decementing of araldited parts is rather uncertain. The limited pot-life of Araldite mixture makes its use somewhat inconvenient in that optical parts being cemented have got to be trued and air bubbles removed before the cement becomes cured/hardened. In this connection it is mentioned that natural resins are also being tried as a substitute for Canada Balsam. Srivastava[6] has reported encouraging results with fresh exudation of *Boswellia serrata* Roxb. (*fam. Burseraceae*), a species growing wild in hotter parts of India.

Araldite is hygroscopic to a little extent and is, therefore, not suitable for use as a bonding medium in a sealed optical instrument in as much as the moisture absorbed at a lower temperature is released at a higher temperature. Its bonding property is, however, excellent and the material can otherwise be used as a general purpose adhesive for bonding glass to metal parts.

CONCLUSION

Araldite, though a good optical cement, is not comparable to Canada Balsam because its decementing is rather uncertain. It is not considered suitable as a bonding material in a sealed optical instrument as it is hygroscopic and therefore causes fogging by releasing moisture at a higher temperature. However, it is a good general purpose adhesive for bonding glass to metal parts.

ACKNOWLEDGEMENTS

Acknowledgements are due to M/s CIBA of India, Ltd., Bombay and Defence Research Laboratory (Materials), Gwalior for helpful suggestions given in the course of the trials. The authors are also thankful to the Director, IRDE, Dehra Dun and M/s CIBA of India Ltd., Bombay for permission to release this paper for publication.

REFERENCES

1. DONALD, E. F., "An Improved Cellulose Caprate Optical Cement" (NRL Report 4242, Washington D.C.).
2. HUNT, P. G., *Optica Acta*, **14** (1967), 401.
3. MEYERHANS, K., *Transac J. Plastics Inst.*, 25 (1957), 267.
4. "CIBA'S Araldite Products", (CIBA Ltd., Basle, Switzerland).
5. TWYMAN, F. "Prism and Lens Making" (Hilger & Watts Ltd., London), 1952, p. 116.
6. SRIVASTAVA, J. G., *Invention Intelligence*, 4 (1969), 71.

APPENDIX 1

QUALITATIVE REQUIREMENTS OF AN OPTICAL CEMENT

(i) Should be almost colourless.

(ii) Should have visible light transmission comparable to that through optical glass.

(iii) Should have low dispersion and refractive index near to that of glass, i.e. between 1·51 and 1·58.

(iv) Should have good adhesion to glass to withstand normal mechanical shocks.

(v) Should be chemically stable and inert to glass.

(vi) Should be homogeneous and free from strain after curing.

(vii) Should have no fluorescence.

(viii) Should not support fungus growth.

(ix) Cemented parts should not deteriorate under varying conditions of temperature and humidity between —40°C and +75°C and upto 100% RH.

(x) Should be resistant to ultra-violet radiation.

(xi) Should be non-toxic and otherwise easy to handle.

(xii) Cementing technique should not be complicated and permit truing operations.

(xiii) Decementing should not be very difficult. [Apparently this requirement is somewhat in conflict with the requirement at (iv). What is desired is that a good compromise should exist between these two requirements].

(xiv) The raw material and cemented parts should not deteriorate in storage.

APPENDIX 2

INSTRUCTIONS FOR PROCESSING ARALDITE

(Supplied by Messrs CIBA of India Ltd., Bombay)

Processing

Surface preparation : The components to be bonded should be cleaned of dirt, grease. oil, etc. adhering to the surface.

Mixing of Resin and Hardener

MIXING RATIOS	PARTS BY WEIGHT	
Araldite AY103	100	100
Hardener HY951	8-9	—
Hardener HY930	—	9-10

Pot-life of the Mixtures

The useable life (pot life) of the mixtures at 20°C and 30°C is as follows :

	AT 20°C	AT 30°C
Araldite AY103 / Hardener HY 951	3 hr.	2 hr.
Araldite AY103 / Hardener HY 930	2½ hr.	1½ hr.

The hardener should not be used more than recommended.

The resin-hardener mixing ratio can also be determined volumetrically. At 20°C, the specific gravity of Araldite AY103 is 1·15 and of Hardeners HY951 and HY930 is about 1.

Curing

The following Table gives minimum curing time at given temperatures based on the mixing ratios recommended.

TEMPERATURE (°C)	(°F)	HARDENER HY951	HARDENER HY930
20	68	36 hr.	24 hr.
40	104	14 hr.	14 hr.
70	158	3 hr.	2 hr.
100	212	1 hr.	1 hr.
130	266	½ hr.	½ hr.
150	302	20 min.	20 min.
180	356	10 min.	10 min.

APPENDIX 3

INSTRUCTIONS FOR DECEMENTING BY USING EPOXY RESIN REMOVER

[Supplied by the Defence Research Laboratory (Materials), Gwalior]

Apparatus Required

400 ml. resistant glass beakers (number as required basis) ; watch glasses to cover the above.

Reagents Required

(i) Epoxy resin remover.

(ii) Concentrated nitric acid (chemically pure grade).

(iii) Acetone (chemically pure grade).

Facilities Required

(i) Fume cupboard with good ventilation arrangement or a suitable hood that is designed and built to serve the same purpose.

(ii) Rubber gloves.

(iii) Mask for protection against acid and corrosive fumes.

Precautions to be Observed

(i) All the operations should be carried out in a fume cupboard or in a suitable hood that is designed and built to serve the same purpose.

(ii) The operator should wear gloves while handling epoxy resin remover. Any accidental spillage of epoxy resin remover should be treated by washing with a reasonably large quantity of rectified spirit. Wash the gloves with plenty of water immediately after use, every time they are used.

(iii) The operator should not allow the acid to come into contact with skin. Any accidental contact should be treated by washing with copious quantities of water. Under no circumstances should the acid be allowed to spurt on to the person of the operator.

(iv) The operator should wear the mask while handling the reagents.

(v) The bottle containing epoxy resin remover should not be kept open and no attempt should be made by any one to inhale or smell this reagent.

Procedure

Keep the lens assembly (made by joining with curing type of Araldite or any similar epoxy resin), that has to be dismantled, in a glass beaker. Pour epoxy resin remover into the beaker to immerse the lens assembly completely. Cover the beaker with a watch glass and set aside at room temperature for 48 hours. Pour out major portion of epoxy resin remover into another beaker and wash the lens assembly with acetone to free it from adhering epoxy resin remover. Wash with water and drain off as much water as possible. Pour concentrated nitric acid to immerse the lens assembly, cover with a watch glass and keep aside at room temperature for 72 hours. Pour out major portion of nitric acid into another beaker and wash the nitric acid adhering to the lens assembly with water. Wash with a little acetone and repeat treatment with epoxy resin remover for 72 hours. Follow this treatment, if necessary, (i.e. if the lens components do not open up by that time) with nitric acid for a further period of 72 hours.

When the lens components open up they are washed with water and finally with acetone and allowed to dry at room temperature for about half an hour and then taken into use.