

## Effect of Constituents and Molecular Structure on Compressive Strength of Cured Epoxide Resins

P. K. Ghosh and A. N. Saha

DEPARTMENT OF APPLIED CHEMISTRY, UNIVERSITY COLLEGE OF SCIENCE AND TECHNOLOGY,  
CALCUTTA-9

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The effect of constituents and molecular structure on compressive strength of cured epoxide resins was studied. It was found that maximum compressive strength was developed in a cured resin block which contained initially maximum amount of diglycidyl ether. The effect of molecular structure of resin on strength showed that alicyclic and aromatic ring substitution in the resin molecule reduces its compressive strength.

The properties of the cured epoxide resin is dependant upon the molecular weight of the parent resin. It is also known from the paper of Walter Erich *et al*<sup>1</sup> that the properties of the cured resin derived from different dihydric phenols is also dependant on the types of the phenols used. These authors did not, however, use dihydric phenols containing alicyclic and aromatic substituents. Moreover, it is not known whether the properties of the cured resin derived from a particular dihydric phenol is dependant, in any way, on the initial amount of diglycidyl ether present in it.

Investigations were made mainly on these two aspects of the problem. The properties of the cured epoxide resins were determined by measuring only the compressive strength of the cured resin block. As the compressive strength of the cured resin block is expected to be a function of the amount of curing agent used<sup>2</sup>, experiments were also conducted to find out the amount of curing agents best suited for curing a particular resin.

### EXPERIMENTAL

Epoxide resins were prepared by condensing 2, 2 bis (4-hydroxyphenyl) propane with epichlorohydrin in three different initial molar ratio viz. 2:1, 4:1 and 10:1. The resins were numbered as (a), (b) and (c). Epoxide resins were also prepared by condensing respectively the bisphenols 2, 2, bis (4-hydroxyphenyl) butane; 1, 1 bis (4-hydroxyphenyl) cyclohexane; and 1-phenyl, 1, 1 bis (hydroxyphenyl) ethane with epichlorohydrin in 10:1 initial molar ratio. The resins were numbered as (d), (e) and (f). The details of the procedure of preparation are described below.



228 gms. of 2, 2 bis (4-hydroxyphenyl) propane and 1000 c.c. of secondary butyl alcohol was placed in a 3-necked reaction flask placed in an oil bath and fitted with a stirrer, a reflux condenser and a charging hole. 82 gms. of powdered sodium hydroxide was added to the flask and the mixture heated to 80-85° for about half an hour to get a clear solution. To this solution was added the requisite amount of epichlorohydrin all at once and the reaction was allowed to proceed for 4 hours at this temperature (80-85°). After the reaction was over, the mixture was mixed with 20 gms. of fullers earth and filtered in a buchner flask to get a clear solution. The solution was then subjected to vacuum distillation to a temperature not exceeding 85° to remove all the sec. butyl alcohol. The resin was obtained as the distillation residue.

Epoxide resins derived from other bisphenols and epichlorohydrin were prepared by condensing similarly 1 mole of the respective bisphenol and 10 moles of epichlorohydrin with 2 moles of Sodium Hydroxide.

The resins were then evaluated by determining the melting point, epoxide equivalent, hydroxyl value, the composition and diglycidyl ether content and also the compressive strength of the cured resin block using ethylene diamine monohydrate as the curing agent.

The epoxide equivalent was determined by pyridinium chloride chloroform reagent<sup>3</sup>. The hydroxyl value was determined by acetyl chloride reagent<sup>4</sup>. The composition and the amount of diglycidyl ether of 2, 2 bis (4-hydroxyphenyl) propane in a resin system containing it, was determined by a thin-layer chromatographic method<sup>5</sup>. The amount of diglycidyl ether of other bisphenols was determined by using diglycidyl ether of bisphenol-A as standard with the assumption that these diglycidyl ethers would behave similarly as that of 2, 2 bis (4-hydroxyphenyl) propane.

In brief, the thin layer chromatographic procedure followed in our study was to elute a 0.3 m.m. thick Silica gel (E. Merck) coated glass plate (10 cm. × 20 cm.) with chloroform. (E. Merck)—the plates were quantitatively spotted with 0.04 ml of a 1% solution of the resin in chloroform with glass capillaries. The elution was done once or twice according to requirements; the dried plates after elution were sprayed with a solution of 50% chromic acid and baked in an oven at 110-120° for a period of about 10 minutes. The spots appeared as bluish black or bluish green on an orange yellow background.

The compressive strength of the cured resins was determined as follows :

About 30 gms. of the accurately weighed resins, heated to a free flowing condition was mixed with the requisite quantity of ethylene diamine monohydrate in a porcelain basin and the mixture was then poured into steel moulds



## RESULTS and DISCUSSIONS

All the resins were liquid except (a) which melted at 60°. The resins (a), (b), (c), (d), (e) and (f) had epoxide equivalents of 483, 353, 191, 202, 216 and 232 and hydroxyl groups per molecule were 2.01, 1.03, 0.16, 0.17, 0.168 and 0.19 respectively.

Fig. 1 and Fig. 2 show the separation of the resin components by thin-layer chromatography and Table 1 gives the % of diglycidyl ether of bisphenols in the resins.

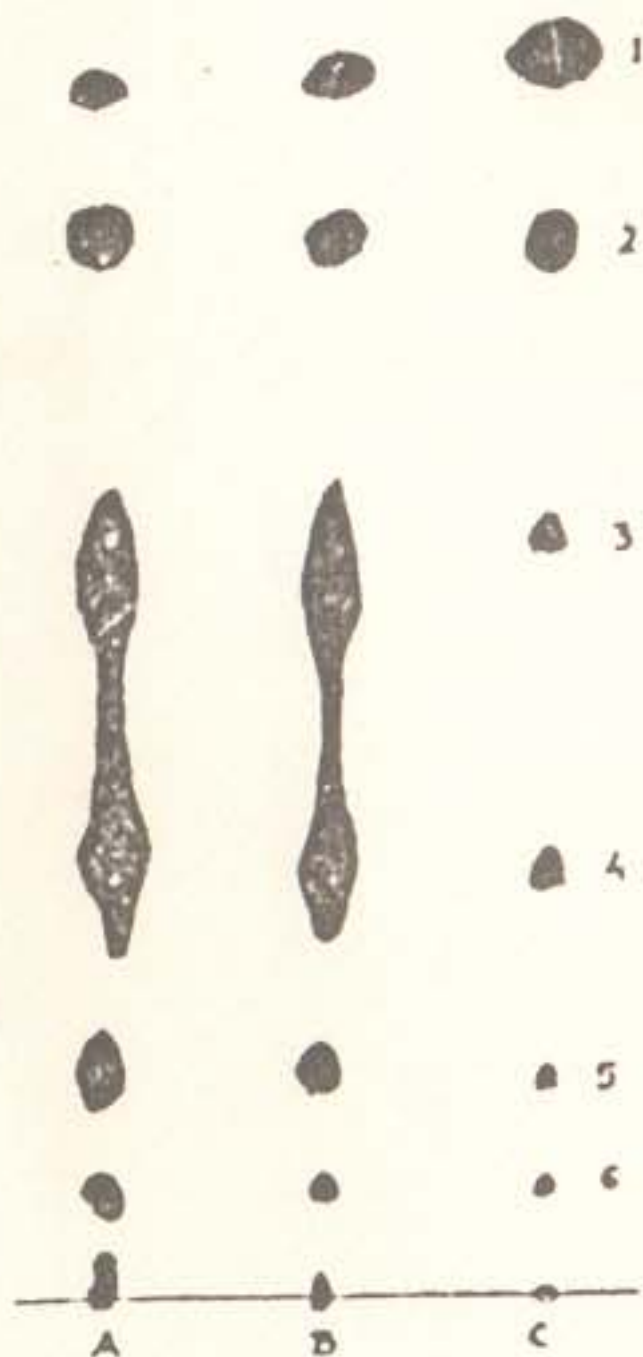


FIG. 1

Thin layer chromatographic separation of the components of resins (a), (b) and (c) spotted at A, B & C respectively. The plates were eluted twice with chloroform and developed by spraying chromic acid and reproduced by tracing the outlines of the spot.

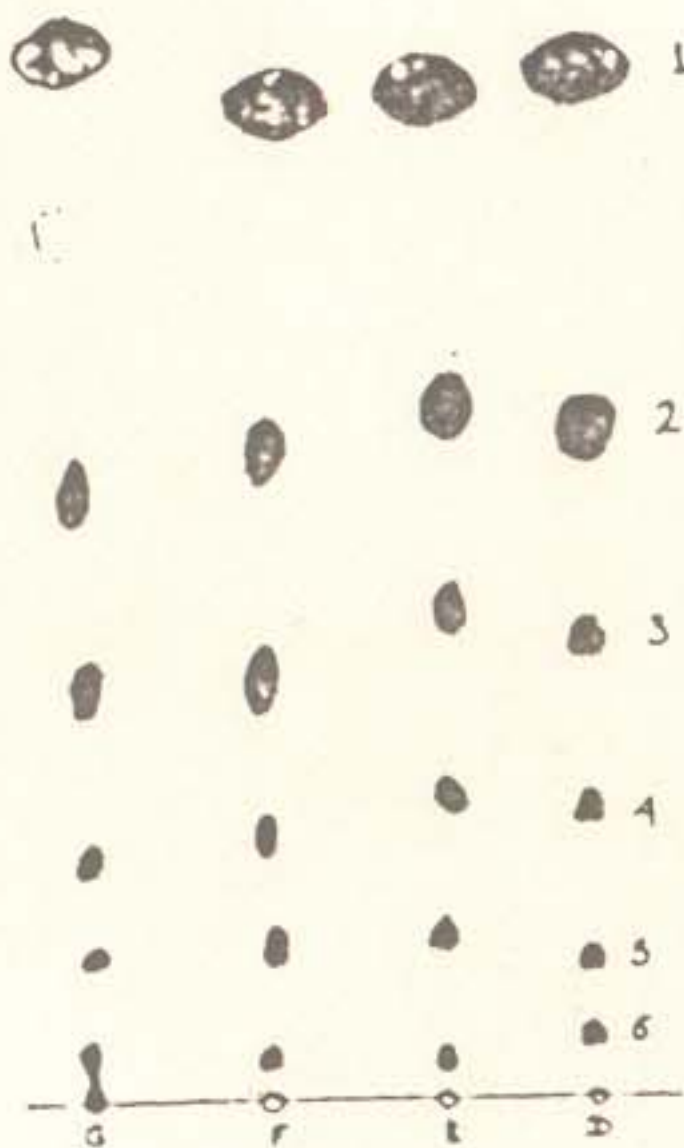


FIG. 2

Thin layer chromatographic separation of the components of resins (c), (d), (e) & (f) spotted at D, E, F & G respectively. The plates were eluted once with chloroform and developed by spraying chromic acid and reproduced by tracing the outlines of the spot.

In both the figures, the spots marked 1 were highly concentric bluish black ones and represented the diglycidyl ethers as was evidenced by comparing their  $R_f$  values with that of pure diglycidyl ether of 2, 2 bis (4-hydroxyphenyl) propane. The other spots marked 2, 3, 4 etc. were diffused ones characterised by a central bluish black portion surrounded by a bluish green shade.



TABLE I

*Diglycidyl ether (%) of bisphenols in the resin*

Resin No.	Diglycidyl ether content (%)
(a)	30-35
(b)	50-55
(c)	85-90
(d)	85-90
(e)	85-90
(f)	80-85

The effect of the amount of ethylene diamine monohydrate on the strength developed in the cured resin is shown in Fig. 3.

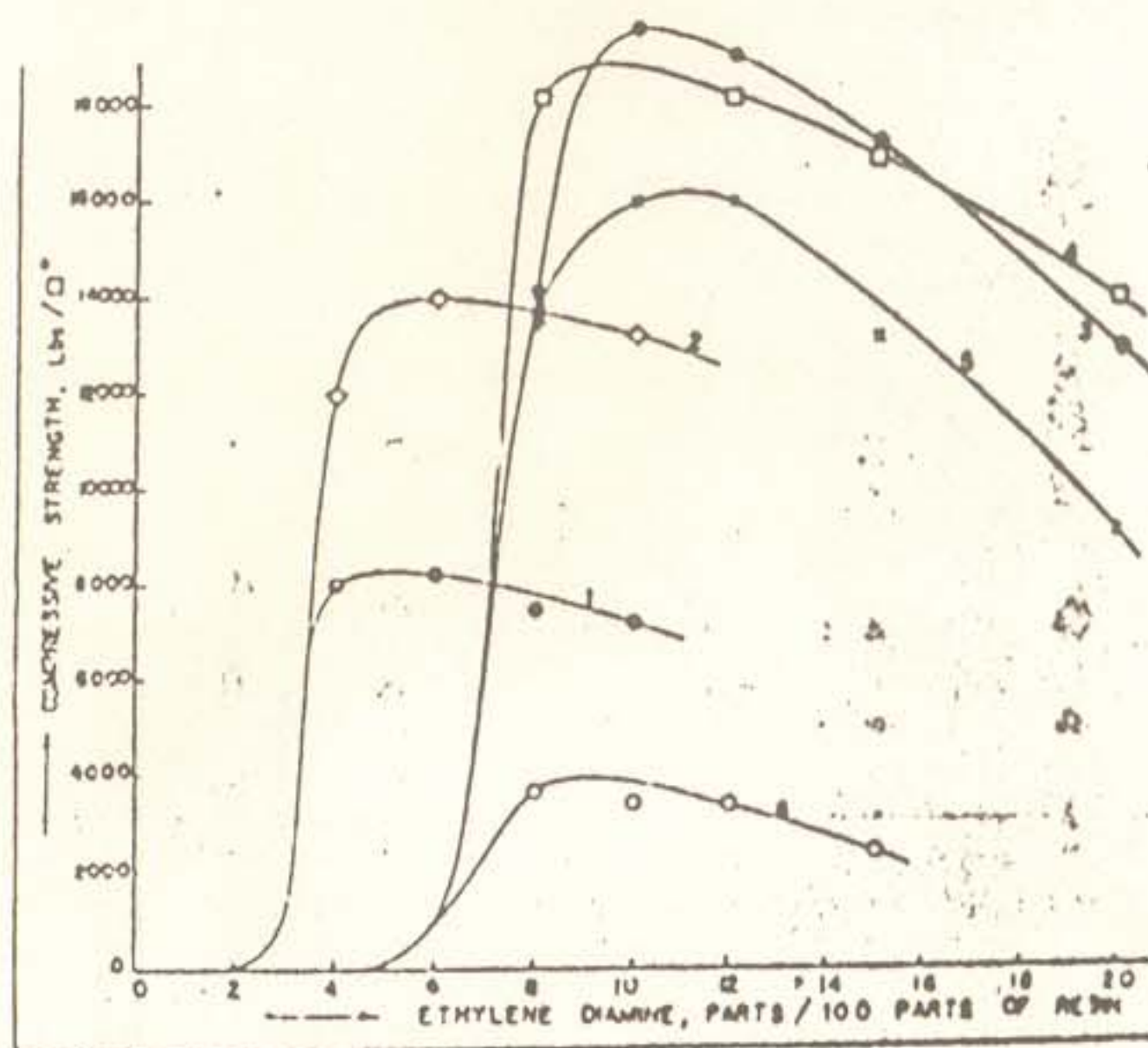


FIG. 3

Effect of the amount of curing agent on compressive strength of cured resin block. Curves marked 1, 2, 3, 4, 5 and 6 were for resins (a), (b), (c), (d), (e) and (f) respectively.

From the data in Table I and Fig. 3 for the resins (a), (b) and (c) it is evident that maximum compressive strength of a cured resin block is dependant on the initial amount of diglycidyl ether present in the resin as is evidenced by the fact that the maximum compressive strength of the cured resins (a), (b) and (c) were 8300, 14000, and 19600 lbs./Q<sup>2</sup> and the diglycidyl ether content was 30-35%, 50-55% and 85-90% respectively.



It is also evident from these data that alicyclic and aromatic ring substitution in the resin reduces its compressive strength. The effect is very much prominent when the substituent group is an aromatic ring. This is evident from the fact that the maximum compressive strength for the resins (c), (d), (e) and (f) were 19600, 18900., 16200 and 4000 lbs| . " respectively.

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