

# Comparison Of The Dynamic Curing Kinetics Of Araldite DLS 772 / 4 4' DDS Epoxy System Using Differential Scanning Calorimetry And Microwave Heated Calorimeter

Babatunde Bolasodun, Alan Nesbitt, Arthur Wilkinson, Richard Day

**Abstract:** - Comparative studies were carried out on the curing kinetics of an epoxy resin Araldite DLS 772 mixed with a hardener, 4 4' Diaminodiphenyl sulfone. Differential scanning calorimetry and a microwave heating calorimeter were used to monitor the curing kinetics of conventionally and microwave cured epoxy samples. These studies were carried out under non-isothermal conditions using five different heating rates. There was a significant increase in the fractional conversion of the microwave cured samples compared to the conventionally cured samples. The curing reactions for samples cured using microwave heating took place over a smaller temperature range. Higher reaction rates were observed in the samples cured using microwave heating. The activation energies of the microwave cured samples of Araldite DLS 772 / 4 4' DDS epoxy system were about ten percent lower than activation energies of conventionally cured samples.

**Keywords-** curing; differential scanning calorimetry; epoxy; microwave heated calorimeter; kinetics

## 1 INTRODUCTION

Epoxy Materials are increasingly being used as high performance structural adhesive systems in the aerospace and the microelectronic industries. Industrially, they are used in the preparation of composites[1]. The extent of cure of epoxy resins has an influence on the physical and mechanical properties; while their processability depends on the rate of polymerization under processing conditions. Thus, kinetic analysis of a resin is very important in understanding the structure-property relationships and for optimizing processing conditions[2]. The increase in demand for epoxy materials has lead to the exploration of faster and more efficient methods of manufacturing epoxy materials [1]. Microwaves have been found to be a good alternative method for curing thermoset polymers. Compared to conventional heating techniques which are based on conduction of heat through a material, microwave heating is a direct form of heating. Microwaves generate heat within the materials. Microwave radiation enables sample temperatures to be potentially changed or controlled more readily [3].

Any increase or decrease in the microwave input power leads to a corresponding increase or decrease in the temperature of the material undergoing cure. Unlike conventional heating which heats the material being processed, along with the walls of the oven and the air surrounding the process material, microwave heating affects only the material being processed. This makes microwave heating a more energy efficient method of heating materials being processed, and this translates into lower production costs for microwave heating [3]. Microwaves do not have any intrinsic difficulties associated with their use, as a result of this, microwave cured products are applied to many diverse industries [4]. There have been numerous studies on the effect of microwave heating on the curing of epoxy resins when compared to conventional heating. Some of these studies report that microwave heating give higher reaction rates[2, 4]; other reports claim that cure proceeds faster with conventional heating[5, 6], while other reports indicate no difference on the effect on microwave heating on the curing of epoxy resins[3]. This article presents the results of the kinetic studies on the conventional and microwave cure of Araldite DLS 772 / 4 4' DDS epoxy system.

## 2. EXPERIMENTAL

### 2.1 MATERIALS

Araldite DLS 772 was the epoxy resin used for this research. It was supplied by Hexcel, UK. Araldite DLS 772 has an epoxy equivalent of 192.33. The hardener used for this study was 4 4' Diaminodiphenyl sulfone. It was supplied by Sigma-Aldrich.

- B. Bolasodun is with the Department of Metallurgical and Materials Engineering, University of Lagos, Nigeria (e-mail: [bbolasodun@unilag.edu.ng](mailto:bbolasodun@unilag.edu.ng))
- Nesbitt is with the National Composites Certificate Evaluation Faculty, School of Materials, University of Manchester, M1 7HS (e-mail: [alan.nesbitt@manchester.ac.uk](mailto:alan.nesbitt@manchester.ac.uk))
- A.Wilkinson is with the National Composites Certificate Evaluation Faculty, School of Materials, University of Manchester, M1 7HS (e-mail: [arthur.wilkinson@manchester.ac.uk](mailto:arthur.wilkinson@manchester.ac.uk))
- R. Day is with Advanced Computer Training and Development Centre, Glydwr University, Wrexham, Wales, LL12 2AW. (e-mail: [r.day@glyndwr.ac.uk](mailto:r.day@glyndwr.ac.uk))

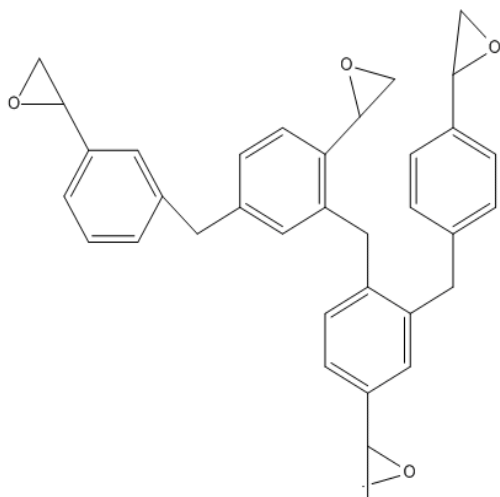


Fig. 1 Chemical Structure of Epoxy Phenol Novalac Resin

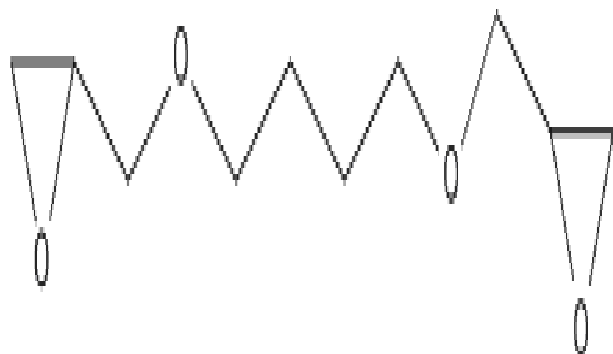


Fig. 2 Chemical structure of 1,4-butanedioldiglycidyl ether

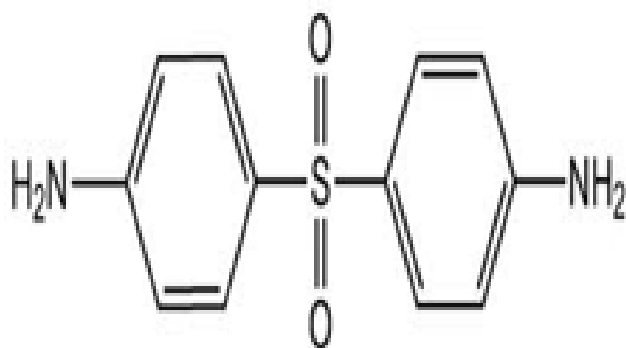


Fig. 3 4,4'-diphenyldiaminosulfone [4]

## 2.2 CURING PROCEDURE

A PerkinElmer Pyris 1 DSC machine was used for the thermal curing of the Araldite DLS 772 / 4,4'-DDS epoxy system. Samples weighing about 3mg were placed in aluminum sample pans; and were cured in a nitrogen atmosphere at dynamic heating rates of 2, 5, 8, 10 and 15°C min<sup>-1</sup>. A microwave calorimeter shown in fig. 1 was used to carry out microwave curing of the samples. The microwave curing was carried out in a cylindrical brass, single mode cavity operating in a TE<sub>111</sub> mode which was designed to give maximum field strength at the centre of gravity. A Hewlett-Packard 8720ET

was used to generate microwaves at a frequency of 2.45GHz; which were then amplified to a maximum power of 30W with a solid state amplifier. An Anritsu power meter was used to measure the transmitted and reflected powers. The power required for heating the sample was calculated by subtracting the transmitted and reflected powers from the amplifier power. This power is similar to the heat flow to the DSC cell in the sense that there will be a decrease in the microwave power during exothermic reaction, while an endothermic reaction will require an increase in the microwave power.

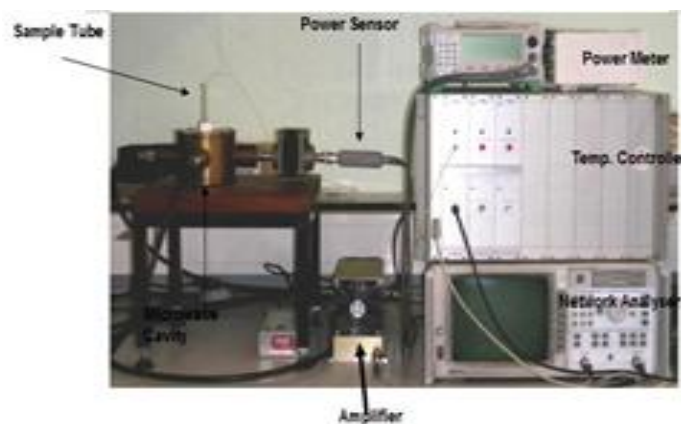


Fig. 4 : Microwave Heated Calorimeter used for this research

For microwave curing, epoxy resin samples weighing about 4g were poured in glass test tubes and placed in the centre of the cavity. A fluoroptic thermometer was inserted in a 1.5 mm diameter glass tube placed in a test tube.

## 2.3 KINETIC ANALYSIS

Epoxy resin curing is an exothermic reaction. For an exothermic reaction, if we assume that [7]

- 1) During curing the exothermic heat generated is proportional to the number of double bonds which have reacted in the system.
- 2) Maximum conversion is attained when all the bonds that can react have reacted.
- 3) The rate of reaction during cure is directly proportional to the rate of heat generation,

At time  $t$ , it becomes possible to determine the degree of conversion  $\alpha$ , and the reaction rate  $\frac{d\alpha}{dt}$  at time  $t$  [15]. This can be determined by the following expressions.

$$\alpha = \frac{\Delta H_t}{\Delta H_R} \quad (1)$$

$$\frac{d\alpha}{dt} = \frac{(dH/dt)_t}{\Delta H_R} \quad (2)$$

Where  $\left(\frac{dH}{dt}\right)_t$  is the rate of heat generation and is directly related to the calorimetric signal at time t, and  $\Delta H_R$  is the total reaction heat associated with the complete conversion of all the reactive groups, and  $\Delta H_t$  is the heat released until time t. This can be obtained directly by integrating the calorimetric signal  $\frac{dH}{dt}$  until the time t [15].

## 2.4 OZAWA'S METHOD

Ozawa's method [8] was used to carry out a kinetic analysis of the epoxy systems. This method is based on isoconversional procedure and it does not require knowledge of the reaction rate equation,  $f(\alpha)$ . Ozawa's method is based on the assumption that regardless of the reaction temperature, the reactive process has the same mechanism of reaction for a given fractional conversion. For the kinetic analysis in dynamic curing experiments, the integral form of the rate equation is expressed as [20];

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\Phi} \int_0^T e^{-(E_a/RT)} dT \quad (3)$$

The value of the right-hand side of equation 8 can be expressed by means of a polynomial function  $p(e/rt)$  as;

$$\frac{E_a}{R} P\left(\frac{E_a}{RT}\right) = \int_0^T e^{-(E_a/RT)} dT \quad (4)$$

Doyle's approximation for the polynomial function  $p(e/rt)$ , which is valid in the range  $20 < e/rt < 60$  is expressed as [9];

$$\log P\left(\frac{E_a}{RT}\right) = -2.315 - 0.4567 \frac{E_a}{RT} \quad (5)$$

In order to determine kinetic parameters in dynamic experiments, Equations 9 and 10 may be combined and rearranged as [10];

$$\log \Phi = \log\left(\frac{AE_a}{g(\alpha)R}\right) - 2.315 - 0.4567 \frac{E_a}{RT} \quad (6)$$

Equation 6 which is also known as Ozawa's method is, for a given degree of conversion, a linear relationship between the logarithm of the heating rate and the inverse of the curing temperature. The activation energy,  $E_a$ , and the constant  $[\log(AE_a/g(\alpha)R) - 2.315]$  can be determined from the slope and the intercept, respectively, of the linear relationship  $\log \Phi$  against  $T^{-1}$  for a constant fractional conversion. For the kinetic analysis of the curing process equation 11 can be applied to different fractional conversion and can also be expressed as;

$$\log \Phi = A' - 0.4567 \frac{E_a}{RT} \quad (7)$$

where  $A' = [\log(AE_a/g(\alpha)R) - 2.315]$ .

## 2.5 KISSINGER'S METHOD

Kissinger's method is another method which is applied to the curing kinetic analysis. [11]. Kissinger's method is based on the assumption that the exothermic peak coincides with the maximum reaction rate and uses an nth-order equation to describe curing kinetics. The expression is described below

$$r = \frac{d\alpha}{dt} = \Phi \frac{d\alpha}{dT} = A \exp\left(\frac{-E_a}{RT}\right) (1-\alpha)^n \quad (8)$$

where r is the reaction rate. Since the maximum rate occurs

when  $\frac{dr}{dt} = 0$ , differentiating Equation 13 with respect to time

and equating the resulting expression with zero gives;

$$\Phi \frac{E_a}{RT_p^2} = An(1-\alpha_p)^{n-1} \exp\left(\frac{-E_a}{RT_p}\right) \quad (9)$$

If we rearrange equation 14, and take the natural logarithms, we get;

$$-\ln\left(\frac{\Phi}{T_p^2}\right) = \ln\left(\frac{E_a}{RAn}\right) - (n-1)\ln(1-\alpha_p) + \frac{E_a}{RT_p} \quad (10)$$

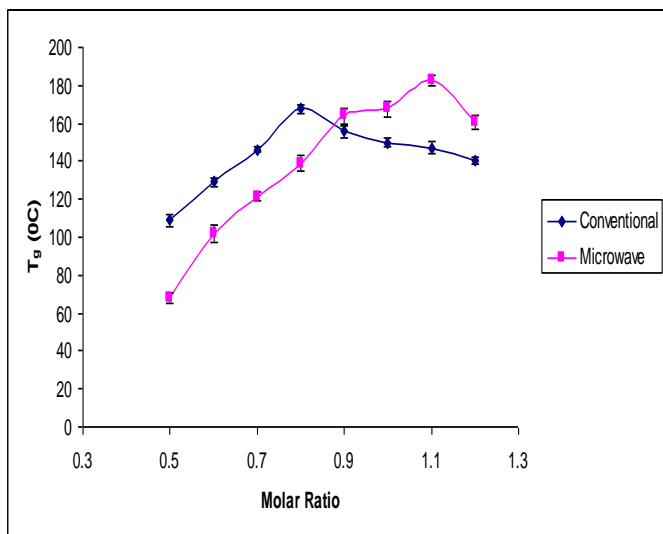
The activation energy can be determined from the slope of the straight line of a plot of  $-\ln(\Phi/T_p^2)$  against  $\frac{1}{T_p}$

## 3 RESULTS AND DISCUSSION

### 3.1 DETERMINATION OF APPROPRIATE STOICHIOMETRIC MOLAR RATIO

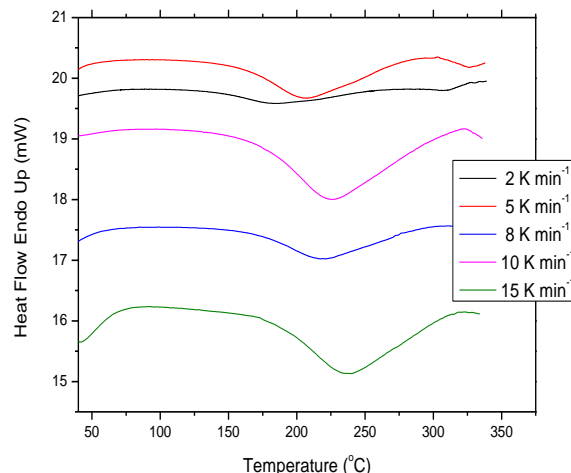
In order to choose the appropriate stoichiometric amine / epoxy (A/E) ratio value to be used for this research, Araldite DLS 772 / 4 4' DDS epoxy systems were prepared with amine / epoxy stoichiometric ratios of 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1, 1.2. DSC scans were carried out on these systems. The systems were heated at  $10 \text{ K min}^{-1}$  from 30 to  $350 \text{ }^\circ\text{C}$  in order to cure the sample. It was then cooled back to  $0 \text{ }^\circ\text{C}$ , and reheated at a heating rate of  $10 \text{ K min}^{-1}$  in order to determine the glass transition temperature value. The epoxy amine / epoxy ratio which gave the highest  $T_g$  was 0.8 and was subsequently used for this research. Similar samples of Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratios of 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1, 1.2 were prepared for microwave curing. A heating rate of  $10 \text{ K min}^{-1}$  was used to cure the sample from 30 to  $310 \text{ }^\circ\text{C}$ , and allowed to cool. Pieces of the cured samples were extracted from the microwave sample tube, and a DSC scan was carried out on the extracted piece from 30 to  $350 \text{ }^\circ\text{C}$  at a heating rate of  $10 \text{ K min}^{-1}$  in order to determine its glass transition temperature. The epoxy amine / epoxy ratio which gave the highest  $T_g$  was 0.8 and was subsequently used for this research. Similar samples of Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratios of 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1 and 1.2 were prepared for microwave curing. A heating rate of  $10 \text{ K}$

$\text{min}^{-1}$  was used to cure the sample from 30 to 310  $^{\circ}\text{C}$ , and allowed to cool. Pieces of the cured samples were extracted from the microwave sample tube, and a DSC scan was carried out on the extracted piece from 30 to 350  $^{\circ}\text{C}$  at a heating rate of 10  $\text{K min}^{-1}$  in order to determine its glass transition temperature.



**Fig. 5** Plot of Glass Transition against Molar ratio for different stoichiometric ratios for both conventional and microwave curing for Araldite DLS 772 / 4 4' DDS epoxy system.

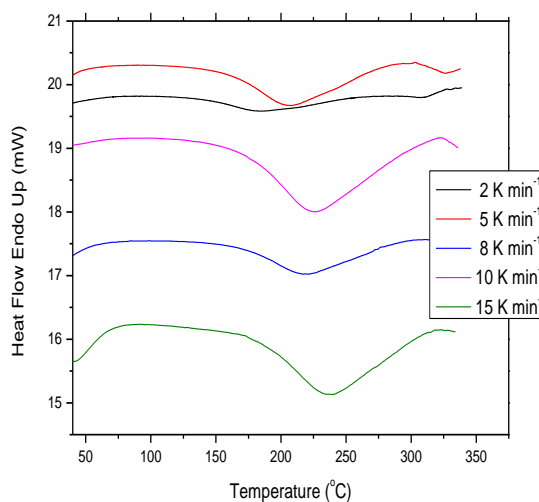
Both plots in fig. 5 show an increase in  $T_g$  values as the Amine / Epoxy ratio increases up to a maximum, and then the  $T_g$  starts to decrease. The highest  $T_g$  values of conventional and microwave heated samples occur at different stoichiometric ratios. The highest  $T_g$  value of the microwave cured sample occurs at a higher Amine / Epoxy Molar ratio than conventional heating. This is an early indication that the curing mechanisms for conventional and microwave curing are different. The typical DSC thermograms for the cure of the Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratio of 0.8 at different heating rates are shown in the fig. 6. It was observed that the temperature at which the exothermic peak occurred depended on the heating rate. The exothermic peak moved towards the right to a slightly higher temperature as the heating rates increased. This is because a thermal lag increases with heating rate, and as a result of this increase, the material starts to react at a higher temperature. The temperature at which the curing reactions were completed also depended on the heating rate. As the heating rate increased, the curing reactions were completed at a higher temperature.

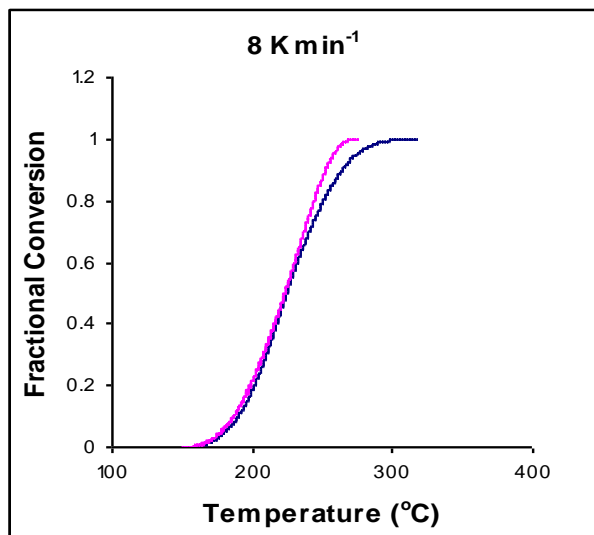
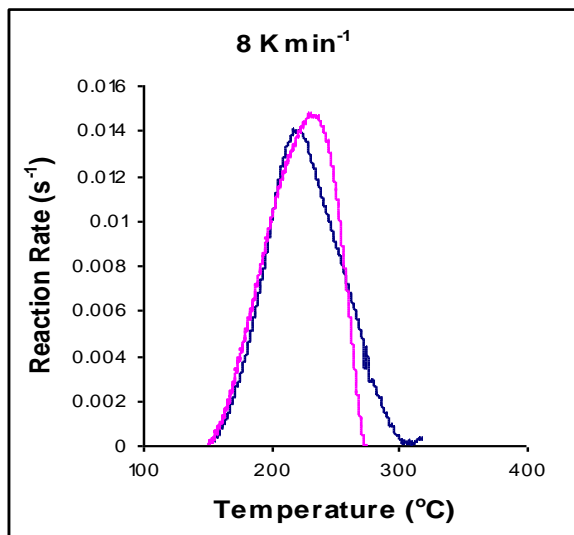
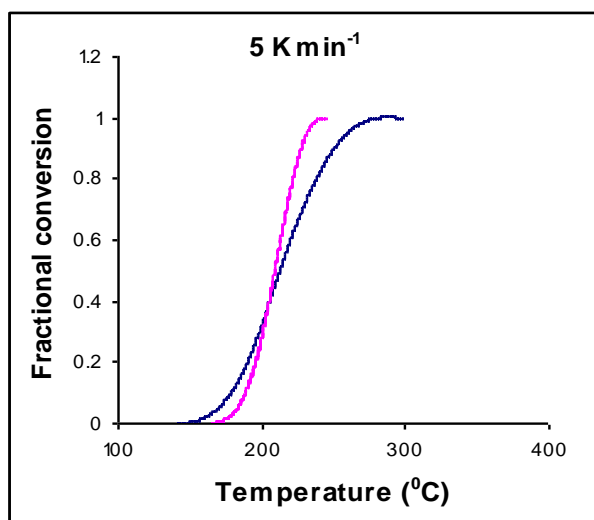
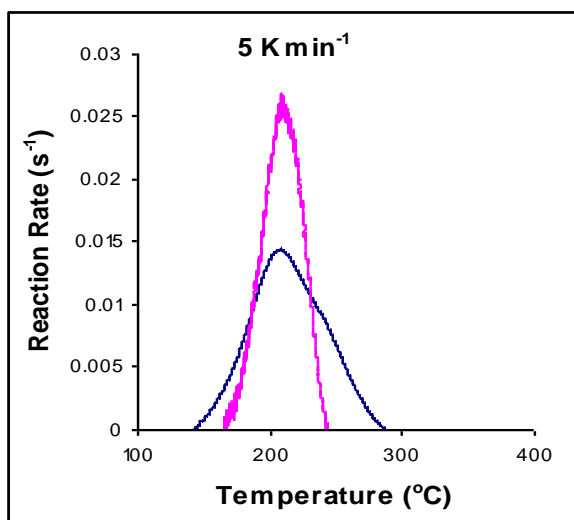
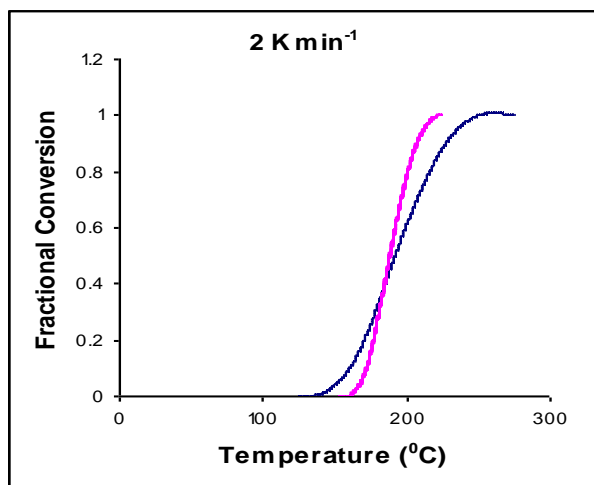
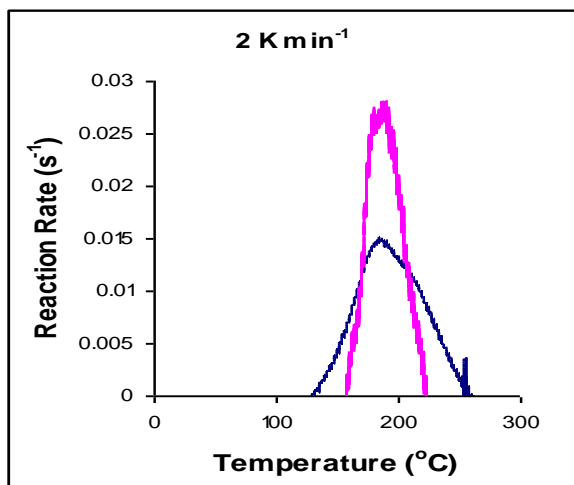


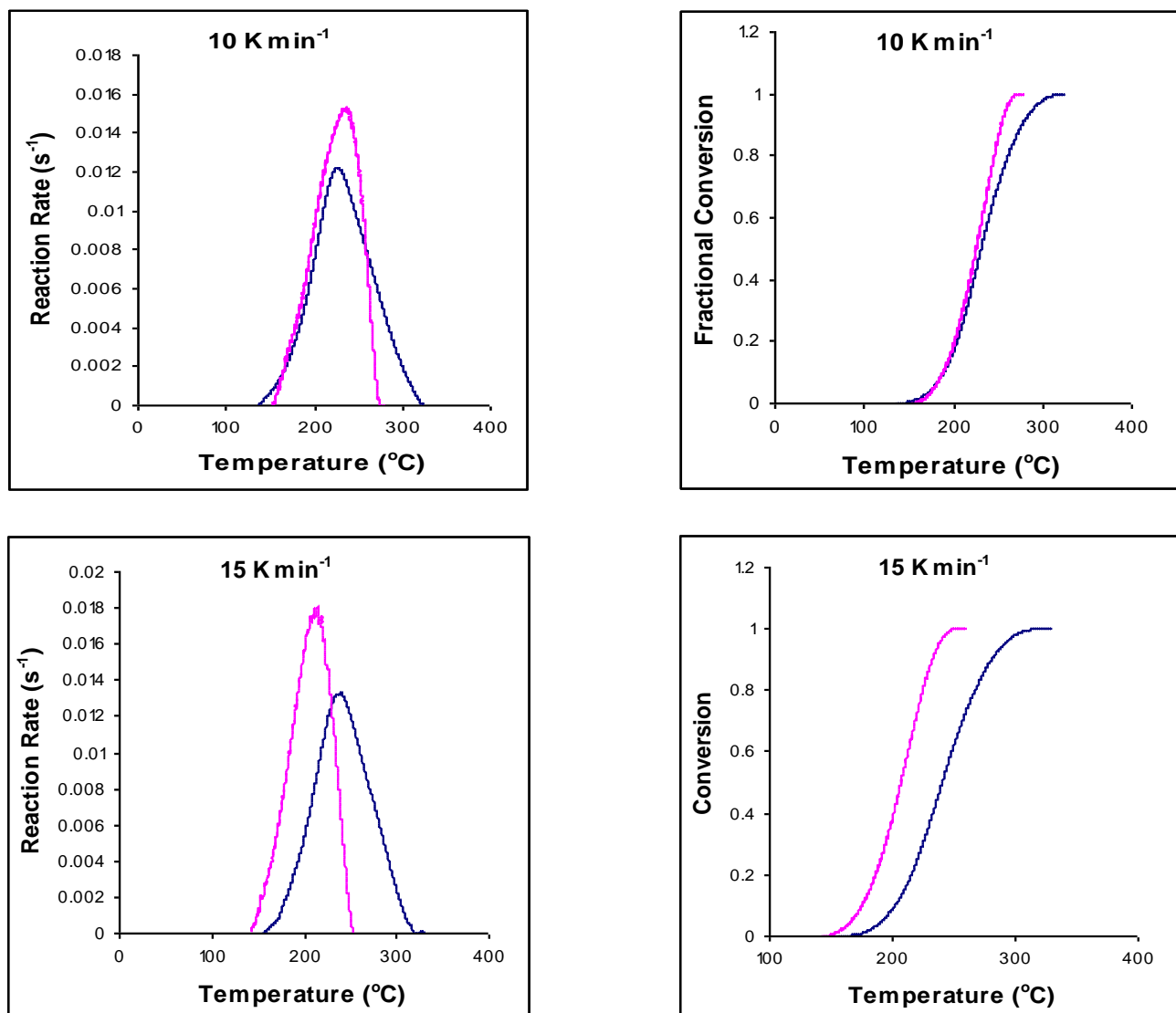
**Fig. 6** Typical DSC thermograms for Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8 at different heating rates using conventional DSC.

### 3.2 CURE KINETICS ANALYSIS

The temperature dependence of the reaction rate and fractional conversion for the curing of Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratio of 0.8 at different heating rates are shown in figure 7 individually. The blue curve indicates the conventionally cured sample, while the red curve indicates the microwave cured sample.







**Fig. 7** Temperature dependence of the reaction rate (left), and fractional conversion (right) for the curing of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8 under conventional (blue) and microwave (red) curing at different heating rates.

**Fig. 7** shows the comparison of the temperature dependence of the fractional conversion and the reaction rates at different heating rates for both conventional and microwave cured Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratio of 0.8. For all the heating rates, the curing

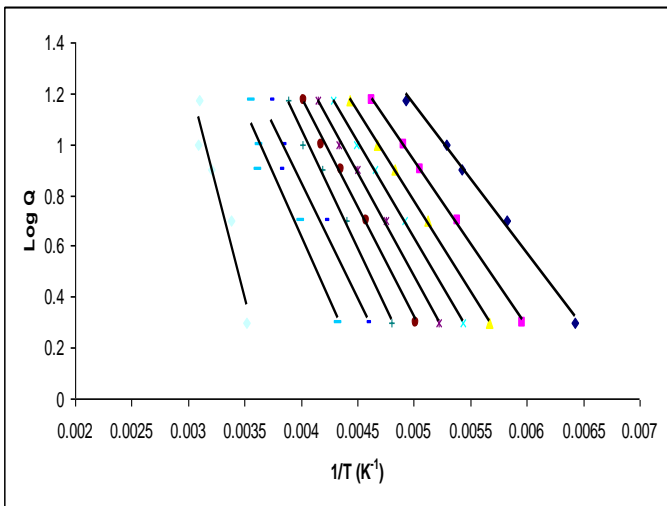
of the Araldite DLS 772 / 4 4' DDS epoxy system with amine / epoxy ratio of 0.8 occurred over a smaller temperature range during microwave curing. The microwave heated samples had a higher peak reaction rate than the conventionally cured samples for all the heating rates used. We also observe that the reaction rate peak occurred at a lower temperature using microwave energy than in the case of conventional. When compared to the conventionally cured samples, there was a significant increase in the reaction rate of the microwave cured samples. For all the heating rates, a higher onset temperature, and a higher reaction rate were observed in microwave cured samples. These differences are all as a result of an improved efficiency in the transfer of energy for the microwave heating. Microwave heating involves the direct delivery of energy to the material as a result of an interaction of molecules with the electromagnetic field. This interaction causes heat to be generated internally throughout the volume of the material [12], whereas in conventional heating, energy is transferred from the surface of the material into the material via conduction or convection. Polymer molecules are heated in the microwave field directly as a result of the relaxation of the dipole polarization along the electromagnetic field. Microwaves are absorbed selectively by the reactive polar molecules, and this greatly enhances the reaction, unlike conventional heating



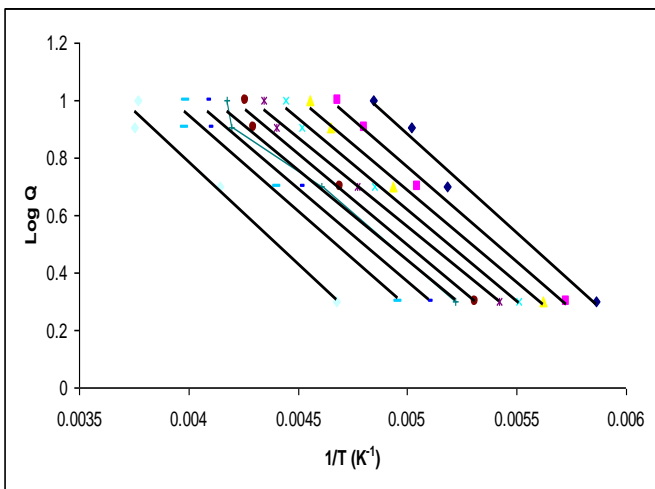
which requires the entire molecule to first be heated before the reaction can take place.[13,14]. The higher fractional conversion for the microwave cured samples can be as a result of an increase in the reactant mobility after gelation. This is as a result of the induced polarization of the polymer and monomer molecules along the applied electromagnetic field [15], enabling more reactants to be consumed to form a more rigid network.

### 3.3 OZAWA'S METHOD

The experimental curves of  $\log \Phi$  against  $T^{-1}$  for various fractional conversions for conventionally and microwave cured Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8 are shown in figs. 8 and 9 respectively.

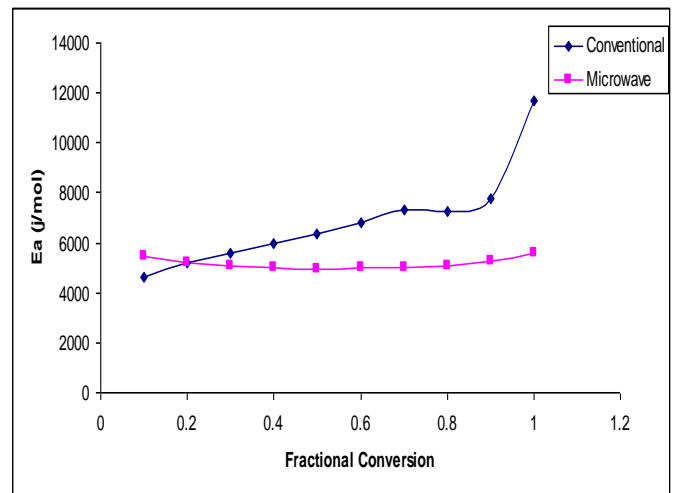


**Fig. 8** Ozawa plots of logarithm of heating rate against the inverse of temperature at constant fractional conversions for the dynamic cure of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8 using conventional heating. Fractional conversions  $\alpha$  of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.00 are shown.



**Fig. 9** Ozawa plots of logarithm of heating rate against the

inverse of temperature at constant fractional conversions for the dynamic cure of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8 using microwave heating. Fractional conversions  $\alpha$  of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.00 are shown in figure 10.



**Fig. 10** Dependence of activation energy,  $E_a$  on the fractional conversion for both conventionally and microwave cured samples of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8.

**TABLE I**  
KINETIC PARAMETERS AT DIFFERENT DEGREES OF CONVERSION FOR CONVENTIONALLY AND MICROWAVE CURED ARALDITE DLS 772 / 4 4' DDS EPOXY SYSTEM WITH AN AMINE / EPOXY RATIO OF 0.8 DETERMINED BY OZAWA'S METHOD

Fractional Conversion ( $\alpha$ )	Activation Energy (E <sub>a</sub> ) Conventional	Activation Energy (E <sub>a</sub> ) Microwave	A' Conventional	A' Microwave
0.1	46.18	54.53	4.07	4.33
0.2	51.89	52.33	4.22	4.08
0.3	55.83	50.69	4.31	3.89
0.4	59.70	50.08	4.41	3.78
0.5	63.69	49.52	4.51	3.70
0.6	68.32	49.94	4.64	3.66
0.7	73.30	49.94	4.77	3.60
0.8	72.39	50.95	4.51	3.59
0.9	77.89	52.49	4.58	3.60
1.0	116.58	55.80	6.45	3.61
<b>Average</b>	68.58	51.63	4.65	3.78
<b>Std. Dev</b>	19.64	4.64	0.67	0.25

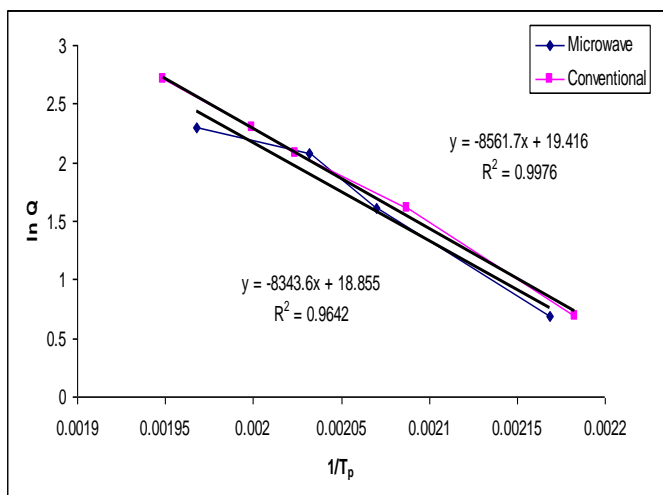
From fig. 10 and Table I, we observe that for conventionally cured samples, the activation energy increases as the fractional conversion increases, while for the microwave cured samples, the activation energy decreases as the fractional conversion increases.

The activation energies and constants A' for epoxy systems are shown in the tables below. In all cases, the regression coefficients obtained were between  $0.98 < r < 1.00$ .

**TABLE II**  
VALUES OF ACTIVATION ENERGY AND PRE-EXPONENTIAL FACTOR FOR ARALDITE DLS 772 / 4 4' DDS EPOXY SYSTEM USING CONVENTIONAL AND MICROWAVE HEATING.

Sample	Conventional Heating		Microwave Heating	
	A' (s <sup>-1</sup> )	E <sub>a</sub> (KJ/mol)	A' (s <sup>-1</sup> )	E <sub>a</sub> (KJ/mol)
Araldite DLS 772 /4 4' DDS	19.42	67.6	18.85	60.9





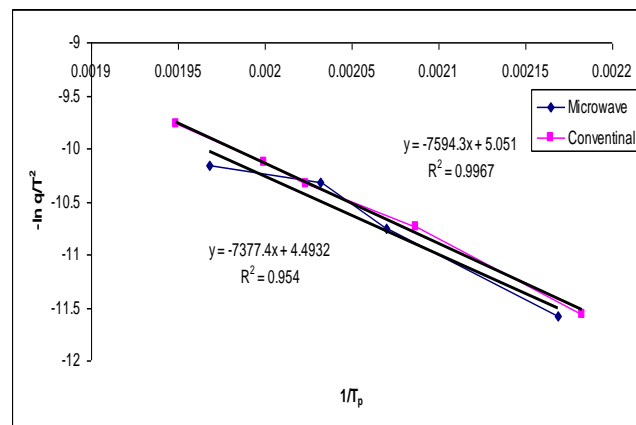
**Fig. 11** Plot of  $\log \Phi$  against  $T_p^{-1}$  for conventional and microwave curing of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8.

From table II, for the Araldite LY 5052 / 4 4' DDS epoxy systems, the activation energies of all microwave-cured samples were higher than those of conventionally cured samples.

### 3.4 KISSINGER'S METHOD

Fig. 12 shows the example of the plots of  $-\ln(\Phi/T_p^2)$  against  $T_p^{-1}$  for the Araldite DLS 772 / 4 4' DDS epoxy system cured using conventional DSC and microwave calorimeter. Interestingly, Kissinger's method gave a value of activation

energy that was similar to the value obtained by Ozawa's method when applied to the exothermic peak. A major difference between Ozawa and Kissinger's method for the determination of activation energy is that in addition to the determination of the activation energy of the entire reaction process, Ozawa's method can be used to determine the activation energy at any specific extent of fractional conversion, while Kissinger's method can only be used to measure the activation energy for the entire reaction process.



**Fig. 12** Plot of  $-\ln(\Phi/T_p^2)$  against  $T_p^{-1}$  for conventional and microwave curing of Araldite DLS 772 / 4 4' DDS epoxy system with an amine / epoxy ratio of 0.8.

**TABLE III**

VALUES OF ACTIVATION ENERGY AND PRE-EXPONENTIAL FACTOR FOR ARLDITE DLS 772 / 4 4' DDS EPOXY SYSTEMS USING CONVENTIONAL AND MICROWAVE HEATING.

Sample	Conventional Heating		Microwave Heating	
	A' (s <sup>-1</sup> )	E <sub>a</sub> (KJ mol <sup>-1</sup> )	A' (s <sup>-1</sup> )	E <sub>a</sub> (KJ mol <sup>-1</sup> )
	5.50	63.1	4.49	59.3

## 4. CONCLUSIONS

The results of the experiments show that for dynamic curing, the temperature at which the reaction rate begins and the exothermic peak temperature are slightly lower in microwave heating. There was an increase in the fractional conversion of the microwave cured samples compared to the conventionally cured samples. The curing reactions for the microwave cured samples took place over a smaller temperature range; and higher reaction rates were observed in the samples cured using microwave heating. Also, Ozawa's method was applied to the exothermic peak for the determination of activation energy. During the non-isothermal curing of the samples, the microwave cured samples of Araldite DLS 772 / 4 4' DDS epoxy system had a lower activation energy than the conventionally cured samples. Kissinger's method was also used to study the kinetic analysis in this research. As with the Ozawa method, the activation energies for the microwave

cured samples were lower than those of the conventionally cured samples for Araldite DLS 772 / 4 4' DDS epoxy system. These lower values have been attributed to the enhancement of the catalytic reaction over the non-catalytic reaction by microwave radiation which occurs as a result of the high activity of the [OH] group in the microwave field.

## ACKNOWLEDGEMENT

This research was carried out at the National Composites Certification and Evaluation Facility, Manchester. Special thanks go to all members of the centre for their support.

## REFERENCES

- [1] Boey, F.Y.C. and B.H. Yap, *Microwave curing of an epoxy-amine system: effect of curing agent on the glass-transition temperature*. Polymer testing, 2001. **20**(8): p. 837-845. H. Poor, "A Hypertext History of Multiuser Dimensions," *MUD History*, <http://www.ccs.neu.edu/home/pb/mud-history.html>. 1986. (URL link \*include year)
- [2] Navabpour, P., et al., *Comparison of the curing kinetics of the RTM6 epoxy resin system using differential scanning calorimetry and a microwave-heated calorimeter*. Journal of Applied Polymer Science, 2006. **99**(6): p. 3658-3668.
- [3] Hill, D.J.T., G.A. George, and D.G. Rogers, *A systematic study of the microwave and thermal cure kinetics of the DGEBA/DDS and DGEBA/DDM epoxy-amine resin systems*. Polymers for Advanced Technologies, 2002. **13**(5): p. 353-362.
- [4] Navabpour, P., et al., *Comparison of the curing kinetics of a DGEBA/acid anhydride epoxy resin system using differential scanning calorimetry and a microwave-heated calorimeter*. Journal of Applied Polymer Science, 2007. **104**(3): p. 2054-2063. D.S. Coming and O.G. Staadt, "Velocity-Aligned Discrete Oriented Polytopes for Dynamic Collision Detection," *IEEE Trans. Visualization and Computer Graphics*, vol. 14, no. 1, pp. 1-12, Jan/Feb 2008, doi:10.1109/TVCG.2007.70405. (IEEE Transactions )
- [5] Costa, M., L. Pardini, and M. Rezende, *Influence of Aromatic Hardeners in the cure kinetics*. Materials Research, 2005. **8**: p. 65-70.
- [6] Mijovic, J. and J. Wijaya, *Comparative calorimetric study of epoxy cure by microwave vs thermal energy*. Macromolecules, 1990. **23**(15): p. 3671-3674. J. Williams, "Narrow-Band Analyzer," PhD dissertation, Dept. of Electrical Eng., Harvard Univ., Cambridge, Mass., 1993. (Thesis or dissertation)
- [7] Salla, J.M. and X. Ramis, *Comparative study of the cure kinetics of an unsaturated polyester resin using different procedures*. Polymer Engineering and Science, 1996. **36**(6): p. 835-851.
- [8] Ramis, X. and J.M. Salla, *Effect of the initiator content and temperature on the curing of an unsaturated polyester resin*. Journal of Polymer Science Part B: Polymer Physics, 1999. **37**(8): p. 751-768.
- [9] Doyle, C.D., *Kinetic analysis of thermogravimetric data*. Journal of Applied Polymer Science, 1961. **5**(15): p. 285-292. J.M.P. Martinez, R.B. Llavori, M.J.A. Cabo, and T.B. Pedersen, "Integrating Data Warehouses with Web Data: A Survey," *IEEE Trans. Knowledge and Data Eng.*, preprint, 21 Dec. 2007, doi:10.1109/TKDE.2007.190746. (PrePrint)
- [10] Ozawa, T., *Kinetic analysis of derivative curves in thermal analysis*. Journal of thermal analysis and calorimetry, 1970. **2**(3): p. 301-324
- [11] Kissinger, H.E., *Reaction kinetics in differential thermal analysis*. Analytical Chemistry, 1957. **29**(11): p. 1702-1706.
- [12] Thostenson, E.T. and T.W. Chou, *Microwave processing: fundamentals and applications*. Composites Part A: Applied Science and Manufacturing, 1999. **30**(9): p. 1055-1071.
- [13] Jacob, J., L.H.L. Chia, and F.Y.C. Boey, *Thermal and non-thermal interaction of microwave radiation with materials*. Journal of Materials Science, 1995. **30**(21): p. 5321-5327.
- [14] Marand, E., K.R. Baker, and J.D. Graybeal, *Comparison of reaction mechanisms of epoxy resins undergoing thermal and microwave cure from in situ measurements of microwave dielectric properties and infrared spectroscopy*. Macromolecules, 1992. **25**(8): p. 2243-2252.
- [15] Wei, J., et al., *Comparison of microwave and thermal cure of epoxy resins*. Polymer Engineering and Science, 1993. **33**(17): p. 1132-1140