Curing of epoxy resins by bio-based phenalkamines vs low-molecular-weight amines: study by DSC

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In this study, curing of BPA-based epoxy resins Araldite®GY-2600 and Araldite®GY-240 by low-molecular-weight amines EDA, TETA and Jeffamine D-230, and bio-based phenalkamines Cardolite®NX-6019, Cardolite®Lite-2002 and Cardolite®GX-6004 was studied by differential scanning calorimetry (DSC) and rheology measurements. DSC provided quantitative information on the overall reaction kinetics (the enthalpy of curing reaction, ΔH_R; cure degree, α; curing reaction rate, $d\alpha/dt$) and the glass transition temperature (T_g) of the cured product. It was demonstrated that the DSC curing of epoxy resins by phenalkamines started at lower temperature, and the curing rates were slightly lower compared to those cured by low-molecular-weight amine hardeners. The enthalpy of the curing by phenalkamines was lower, especially in the case of more viscous epoxy resin GY-2600. T_g of the cured epoxy resins varied from 50 to 98°C and was slightly lower when cured with cardanol-based phenalkamines. The results demonstrate that curing of epoxy resins by bio-based phenalkamines proceeds in a similar temperature interval like using low-molecular-weight amines as hardeners but with less exothermic effect, which could be an advantage.

**Keywords:** phenalkamines, epoxy resins, curing reaction, *pot-life*, DSC

INTRODUCTION

Phenalkamines (PA) are a relatively new class of curing agents for epoxy resins, widely used in protective coatings. Advantages of these curing agents are related to low/room temperature curing applications, a good moisture tolerance and a good surface appearance of the coatings [1–3]. PA are bio-based amines synthesised from cardanol refined from renewable material, Cashew nut shell liquid (CNSL) [3, 4]. CNSL has proven itself to be an invaluable resource for the development of a wide variety of products including not only epoxy curing agents but also other components of epoxy resins and polyurethanes, and much more [5].

Phenalkamines are Mannich base generally synthesised from equimolar quantities of cardanol, formaldehyde and polyamine (Scheme 1) [6].

The distinctive structure of PA has some effect on the properties of the resulting paints and coatings. Due to a long aliphatic alkyl side chain of cardanol, PA-containing paints are characterised by lower viscosity and surface tension, and improved wetting properties; the resulting coatings possess a good flexibility and hydrophobicity, and an improved water resistance. The aromatic backbone of PA gives an excellent chemical resistance. Side chains of PA containing amino- and imino-groups react easily with the epoxy groups giving highly cross-linked products. A high activity of PA even at ambient temperature as well as a good adhesion is related to the presence of the phenolic

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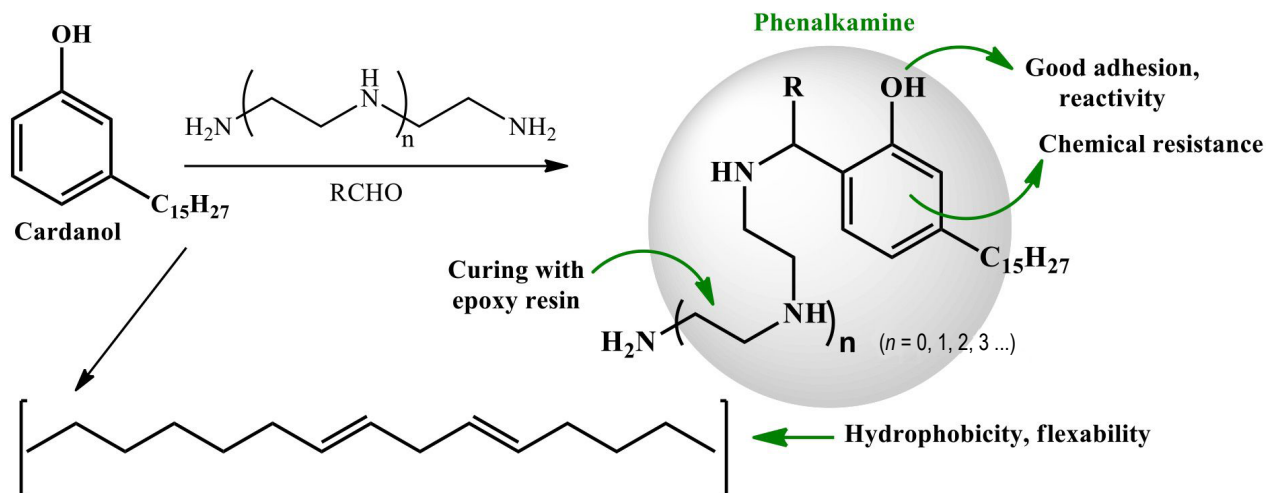
Scheme 1. The synthesis and structure–property relationship of phenalkamines [6]

hydroxyl group. Furthermore, PA hardeners are known for a low toxicity, which makes them eco-friendly.

Curing low-molecular-weight epoxy resins by amine hardeners, tightly cross-linked materials with high T_g are formed usually [7]. To reduce the brittleness (and the same, T_g) of the cured epoxy resins, phenalkamines can be successfully used in an eco-friendly way. The curing mechanism and properties of the epoxy coatings depend on the PA structure, molecular weight and amine value.

Differential scanning calorimetry (DSC) is a thermal analysis technique widely used to obtain a thorough understanding of the cure kinetics of various systems. A DSC calorimeter captures the exothermic heat flow which is released during the cross-linking process of a reactive polymer in respect of curing time under isothermal or non-isothermal conditions [8]. The curing kinetics of various epoxy-based systems has been extensively investigated by this method [9–15]. Furthermore, DSC is very useful to gain additional information about the extent of cross-linking by changes in glass transition temperature (T_g). Some authors declare that T_g is even more suited for monitoring of the cross-linking process than the cure degree α [16, 17].

Rheology is the science of deformation and the flow of matter and describes the interrelation between force, deformation and time. Flow properties of a fluid are usually characterised by a plot of the shear stress associated with the shear rate applied to the fluid. One of the important rheological terms is viscosity, which is a measure of its resistance to deformation at a given rate. The initial viscosity of epoxy resin–amine mixture is increasing with curing time, which could serve as an indicator of the curing rate [18–20].

The objective of the present work was to evaluate the kinetics of DSC curing of commonly used liquid epoxy resins by bio-based phenalkamines and compare with curing using typical low-molecular-weight amines. The further objective was to identify the specific features of curing of epoxy resins by cardanol-based phenalkamines in regard with the thermal effect, development of cure degree, and *pot-life* of the curing systems.
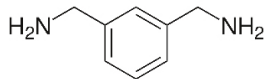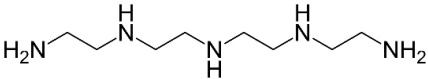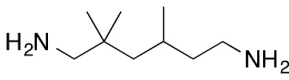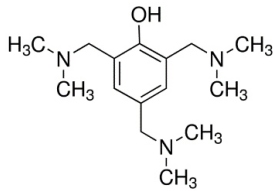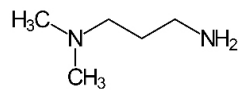
EXPERIMENTAL

Materials

Commercial epoxy resins and amine hardeners typically used in anti-corrosion epoxy-based coatings were used in this study. Bisphenol-A epoxy resins with commercial names Araldite® GY-240 (epoxy equivalent weight (EEW) 178–183 g/eq, viscosity (25°C) 7000–9000 mPa·s) and Araldite® GY-2600 (EEW 184–190 g/eq, viscosity (25°C) 12000-14000 mPa·s) were from Huntsman Co. Low-molecular-weight amines 1,2-ethanediamine (EDA, amine equivalent weight (AEW) 15 g/eq), triethylenetetramine (TETA, mixture of four ethyleneamines with similar boiling points including linear, branched and two cyclic, AEW = 24 g/eq) and amine-terminated polyoxypropylene glycol

(polyetheramine) with the commercial name Jef-famine® D-230 (AEW = 60 g/eq) were provided by Huntsman Co. Cardanol-based phenalkamines with the commercial name Cardolite® were kindly donated by Cardolite Co. The commercial names and the composition of bio-based phenalkamines are given in Table 1.

Batches for DSC measurements were made by mixing 1 g of epoxy resin and the corresponding amount of an amine hardener. The mixing ratio of epoxy resin to a curing agent was stoichiometric. The amount of a hardener (g) required to cure 1 g of an epoxy resin was calculated by the equation [21]:

Table 1. **The composition of cardanol-based commercial phenalkamines***

Components	Cardolite®		
	Lite-2002, % (AEW = 104 eq/g)	GX-6004, % (AEW = 76 eq/g)	NX-6019, % (AEW = 133 eq/g)
Cardanol-based phenalkamine	72–78	50–62	68–75
1,1′-(1,3-Phenylene)di(methanamine) *m*-Xylylenediamine (MXDA)	18–22	15–23	1–2
Cashew (*Anacardium occidentale*) nutshell extract, decarboxylated, distilled	–	16–20	18–21
N¹-(2-Aminoethyl)-N²-{2-[(2-aminoethyl)amino]ethyl} ethane-1,2-diamine Tetraethylenepentamine (TEPA)	4–6	–	–
2,2,4-Trimethylhexane-1,6-diamine (TMD)	–	~7	–
2,4,6-Tris[(dimethylamino)methyl]phenol (DMP)	–	–	3–4
N¹,N¹-Dimethylpropane-1,3-diamine Dimethylaminopropylamine (DMAPA)	–	–	2–3
Phenol	–	–	0.6–0.9
Ethylenediamine (EDA)	–	–	0.6–0.9

* From the material safety data sheets (MSDS) of phenalkamines.

$$m_h = \frac{\text{AEW} \cdot m_{\text{epoxyresin}}}{\text{EEW}}.$$

The general structure of BPA-based epoxy resins and amines used in this study is presented in Scheme 2.

DSC measurements

A calorimeter Perkin Elmer DSC8500 was used to study curing of epoxy resins by amines. A non-isothermal DSC test was performed under nitrogen atmosphere (rate 40 mL/min). A sealed empty aluminium pan was used as a reference. Samples of 3–5 mg were heated from –60 to 200°C at the rate of 10°C/min. and then kept at this temperature for additional 10 min. The heat flow curves were recorded, and the thermal data were further processed to determine the cure degree (α) and the curing reaction rate ($d\alpha/dt$). After the first heating, the temperature was decreased to –60°C and equilibrated for 3 min. Then the second heating at the rate 10°C/min to 200°C was performed, which was used for determination of the glass transition temperature (T_g) of the cured system.

Rheological measurements

A rheometer Anton Paar MCR302 was used to study the initial stage of curing and for the determination of *pot-life* of the curing system. The *pot-life* refers to the amount of time it takes for the product's initial mixed viscosity to double [22]. The measurements were performed at 25°C with a parallel-plate of diameter 50 mm and a distance between the parallel plate and the base

mount of 1 mm, and the shear rate was kept constant at 0.25 s^{-1}. The viscosity (in mPa·s) was measured every 5 s.

RESULTS AND DISCUSSION

To demonstrate the curing progress of the epoxy resin–amine system, non-isothermal DSC tests were carried out. Some results of these tests presented in the form of DSC curves are shown in Fig. 1. The heat flow as a function of temperature was determined and the thermal data were further processed to determine the cure degree α (Fig. 2) as well as the rate of the curing reaction $d\alpha/dt$ (Figs. 3–4). The cure degree α was calculated by the ratio $\alpha = \Delta H_t/\Delta H_T$ [23], where ΔH_t represents the enthalpy of the curing reaction at time t, and ΔH_T is the total enthalpy of the curing reaction of the epoxy resin. ΔH_T was calculated by integrating the heat flow over the whole exothermic peak for a completely uncured sample. The curing reaction rate ($d\alpha/dt$) is proportional to the measured heat flow (dH/dt) [24] or, the same, to the changes in the cure degree. $d\alpha/dt$ was calculated by taking the derivatives of the curves presented in Fig. 2.

Evidently, the curing reaction is an exothermic process which is represented by one exothermic peak (Fig. 1). From the curing curves, the initial curing temperature (T_i), peak curing temperature (T_p), terminal curing temperature (T_t) [25] and enthalpy of the curing reaction (ΔH_R) were obtained and listed in Table 2. Some data were not processed due to the instability of the baseline (cases of EDA and TETA). The initial curing temperature T_i did not depend on the type of the epoxy resin
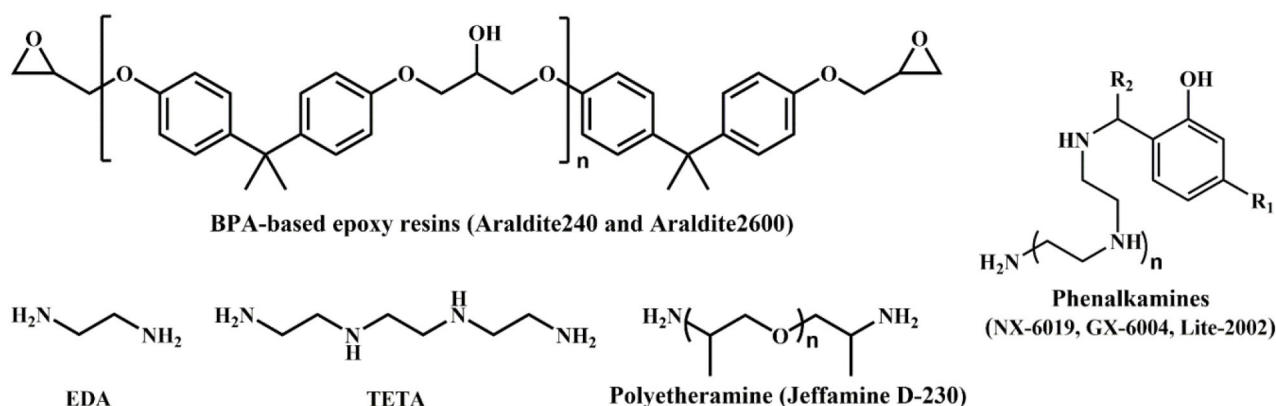
Scheme 2. Structure of the epoxy resins and amine hardeners used in the present study
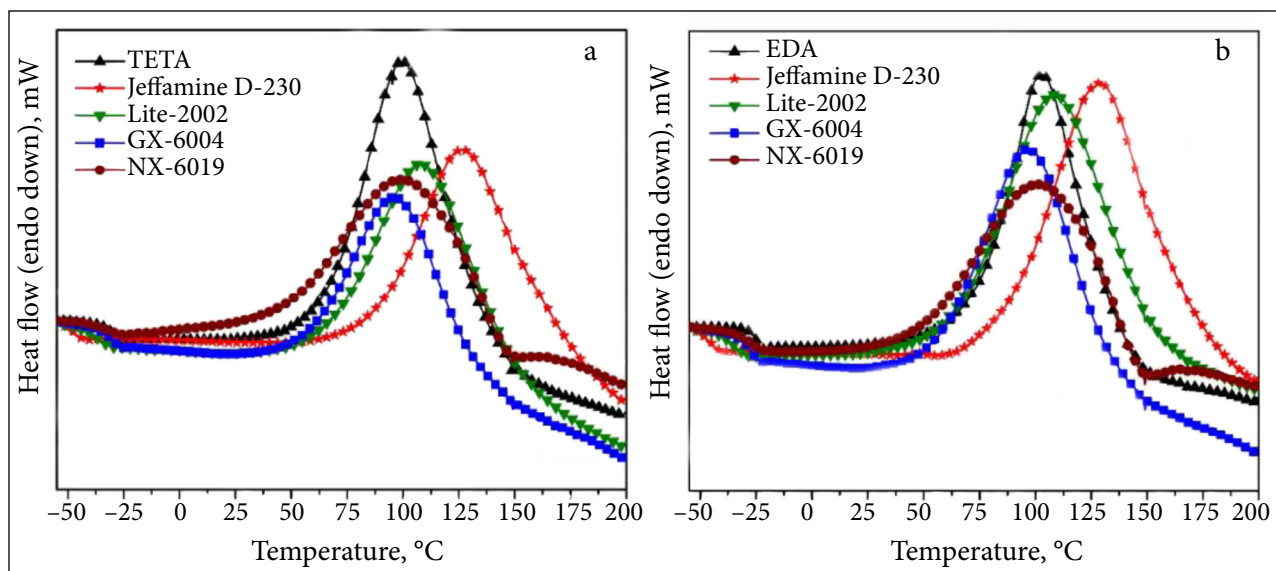
Fig. 1. DSC curing of the epoxy resins GY-240 (a) or GY-2600 (b) by different amines

but was dependent on the type of the hardener being the lowest for the phenalkamines NX-6019 and GX-6004 (Table 2). Curing by Jeffamine D-230 was evidently late, which was reflected in higher values of T_i, T_p and T_t. The peak curing temperature T_p obtained by using other hardeners including phenalkamines was similar and close to 100°C.

During curing, the cure degree α starts at 0 and can reach a maximum value of 1, representing the fully cured epoxy resin (Fig. 2). The relationship between the curing reaction rate ($d\alpha/dt$) and temperature is presented in Fig. 3. The curing rate in all the cases increases gently when the epoxy resin–amine system begins to cure. As the reaction progresses, the heat released by the curing reaction itself simultaneously accelerates the curing process. Thus, the internal system epoxy resin–amine most likely exhibits an autocatalytic curing reaction mechanism (Scheme 3) [9].

The maximal values of the curing reaction rate were reached at about 100°C with an exception for the mixture containing Jeffamine D-230. When most of the functional groups were reacted, the curing rate of the epoxy resin–amine system started to gradually decrease and at 150–200°C approached the values close to 0.
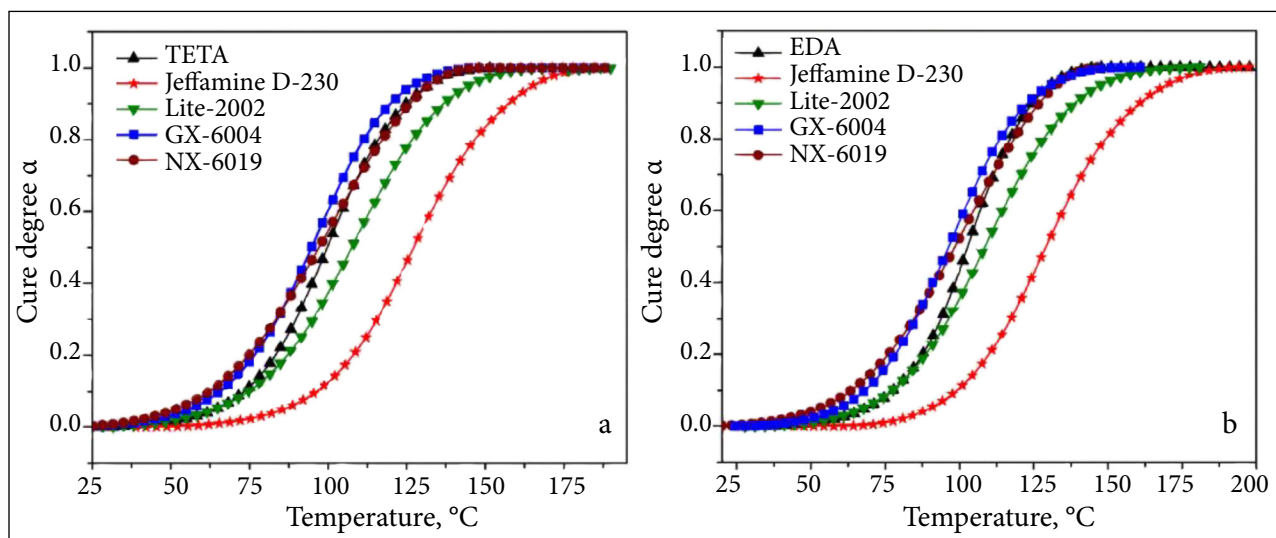

Fig. 2. The dependence of the cure degree α on temperature under DSC curing of the epoxy resins GY240 (a) and GY2600 (b) by different amines

Table 2. **Characteristic parameters of the DSC curing of epoxy resins by different amines**

Amine hardener	T_i, °C	T_p, °C	T_t, °C	$\Delta T = T_t - T_i$, °C	ΔH_R, J/g	T_g
Epoxy resin GY-240						
EDA	72.6	100.9	132.7	60.1	–	76
TETA	64.7	100.0	142.7	78.0	431.4	–
Jeffamine D-230	85.2	126.5	188.9	103.7	386.0	80
Lite-2002	64.2	108.9	161.3	97.1	363.9	73
NX-6019	48.2	99.9	146.0	97.8	344.9	50
GX-6004	55.0	96.8	136.0	81.0	308.5	85
Epoxy resin GY-2600						
EDA	71.5	103.1	141.4	69.9	312.4	53
TETA	62.9	94.7	142.4	79.5	–	98
Jeffamine D-230	83.2	127.7	186.1	102.9	397.9	71
Lite-2002	63.9	108.9	158.0	94.1	335.1	76
NX-6019	47.1	103.0	146.9	99.8	276.8	53
GX-6004	55.5	96.8	137.2	81.7	291.4	82

The relationship between the curing rate $d\alpha/dt$ and the cure degree α is given in Fig. 4. As the curing reaction progresses, the curing rate increases, reaches maximal values at the degree of curing 0.45–0.55 and then gradually decreases. There are no obvious differences in curing by low-molecular-weight amines and bio-based phenalkamines.

Enthalpy of the cure reaction of the epoxy resin GY-240 by various hardeners was slightly higher compared to that of GY-2600. Curing of the epoxy resins by low-molecular-weight amines TETA and Jeffamine D-230 was accompanied by the highest exothermic effect (ΔH_R about 386–431 J/g). The highest curing rates (more than 0.2 min^{-1}) were characteristic of the epoxy resins cured by low-molecular-weight amines EDA and TETA. Curing by

these hardeners started at 63–73°C, and the cure interval was relatively narrow and ranged 60–80°C. Surprisingly, curing of the epoxy resins by poly-etheramine Jeffamine D-230 proceeded at a significantly higher temperature. The curing rate of this system was lower (about 0.175 min^{-1}), irrespective of the epoxy resin used, and the full cure was reached at much higher temperature (at about 190°C).

The enthalpy of curing of the epoxy resins by phenalkamines Lite-2002, GX-6004 or NX-6019 was lower (ΔH_R 277–364 J/g). In these cases, the cure process started earlier (at about 47–64°C), and the cure interval was broader (about 80–100°C). The lowest curing rates were characteristic of the epoxy resins mixed with phenalkamines NX-6019 and Lite-2002, 0.15 min^{-1} and 0.175 min^{-1}, respectively (Fig. 3).
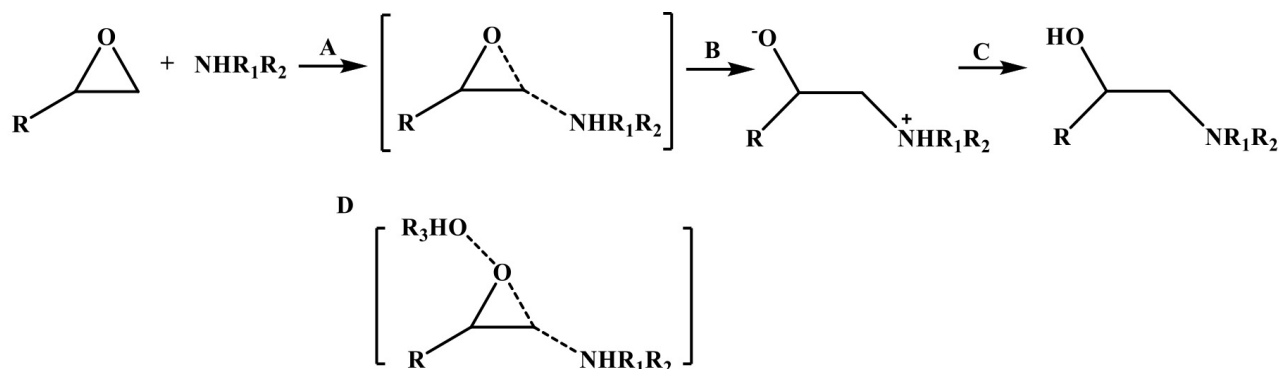
Scheme 3. The general mechanism presenting the autocatalytic nature of curing epoxy resins by amines: A, formation of an intermediate complex; B, ring-opening; C, proton transfer; D, the autocatalytic role of OH groups
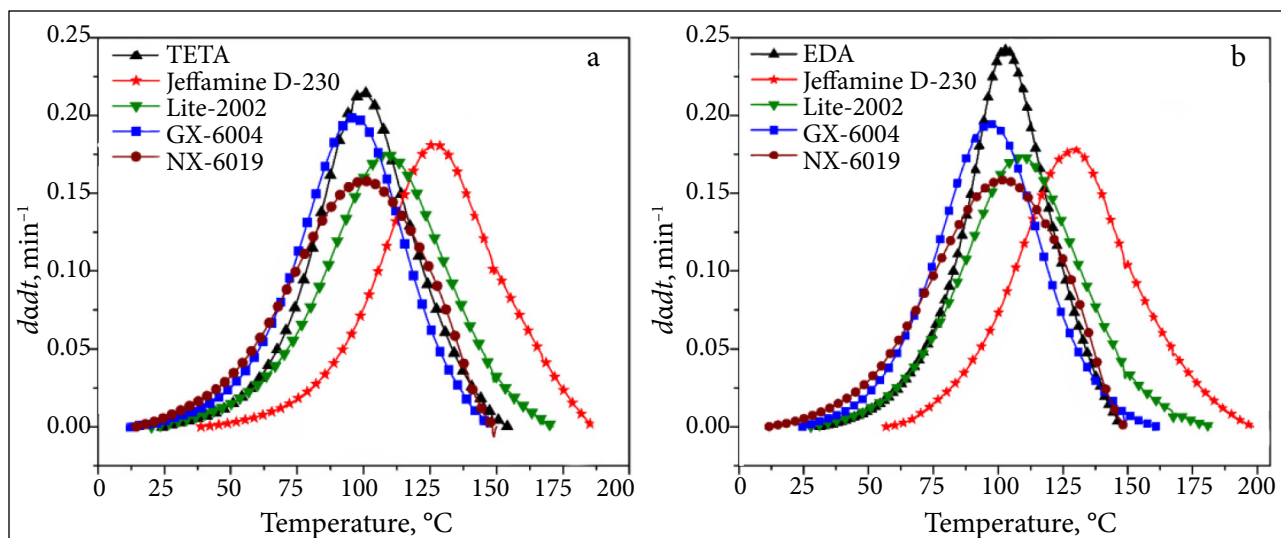
Fig. 3. The dependence of the curing reaction rate $d\alpha/dt$ on temperature under DSC curing of epoxy resins GY-240 (a) and GY-2600 (b) by different amines

Curing by the phenalkamine GX-6004 proceeded a little bit faster ($d\alpha/dt$ almost 0.2 min⁻¹) giving the lowest peak curing temperature (T_p 96.8°C). Thus, it seems that GX-6004 is the most active phenalkamine among all investigated. This phenomenon can be attributed to highly reactive 2,2,4-trimethylhexane-1,6-diamine (TMD) [26] present in the composition of this phenalkamine.

The glass transition temperature (T_g) of the cured epoxy resins is associated with the crosslinking density of the system (Fig. 5). T_g of the cured epoxy resins varied from 50 to 98°C and was dependent on the type of hardener (Table 2). The effect of the type

of the epoxy resin (GY-240 or GY-2600) on T_g was much less. The highest T_g (98°C) was characteristic of the epoxy resin GY-2600 cured by TETA. Likely, a tightly crosslinked network was formed in this case due to a high functionality of TETA and short distances between active centers. Lower values of T_g were characteristic of the epoxy resins cured by EDA and Jeffamine D-230. The crosslinking density in the cured epoxy resins is expected to be lower in these cases due to a lower functionality of these hardeners. Lower T_g of the epoxy resins cured by phenalkamines NX-6019 and Lite-2002 (50–76°C) could be attributed to the long alkyl chains present
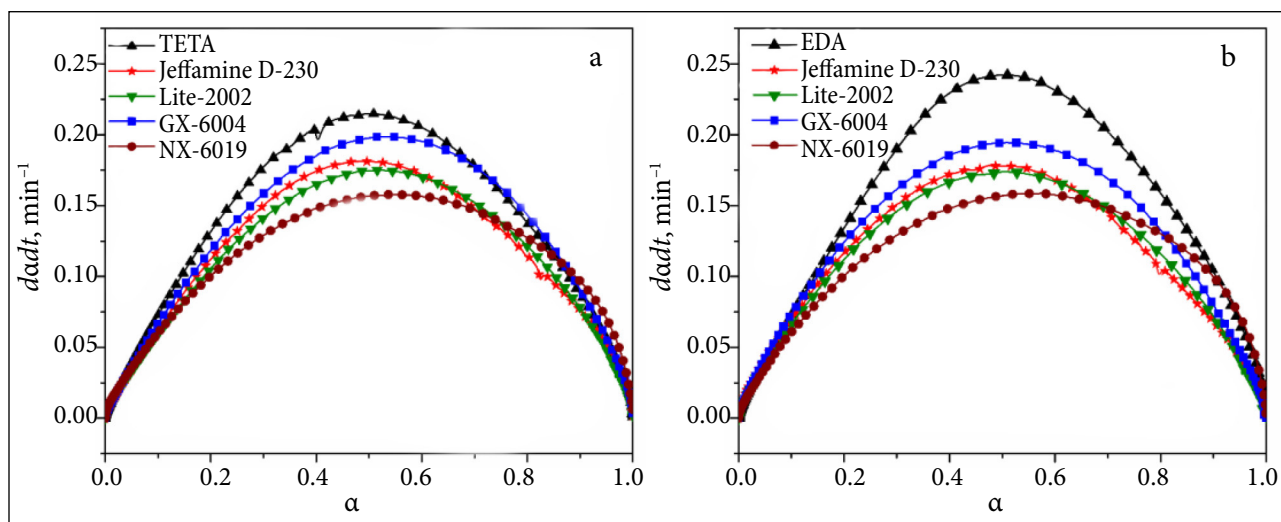

Fig. 4. The relationship between the curing rate $d\alpha/dt$ and the cure degree α under DSC curing of epoxy resins GY-240 (a) and GY-2600 (b) by different amines
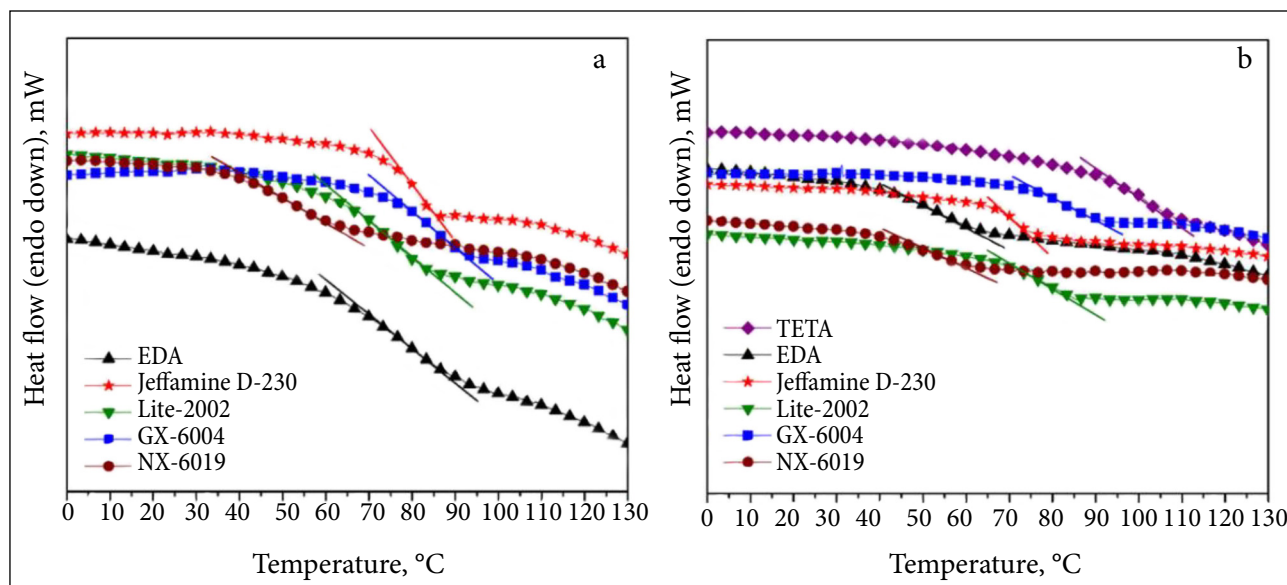
Fig. 5. DSC curves of the epoxy resins GY-240 (a) and GY-2600 (b) cured using different amines (second heating)

in the phenalkamines, which increase the flexibility and chain mobility in the cured system. Slightly higher T_g (82–85°C) was characteristic of the epoxy resins cured by phenalkamine GX-6004. This correlates well with a higher reactivity of this hardener demonstrated by DSC studies.

The curing of epoxy resins by amine hardeners results in a consistent increase in the viscosity of the system. The rate of viscosity changes could be an indicator of the activity of reacting components during the initial stage of curing. Changes in the viscosity of the curing epoxy resins at room temperature during 1–2 h after mixing were evaluated by a rotational rheometer. The *pot-life* of the curing systems was considered to be a quantitative parameter reflecting the rate of the cure process (Table 3). The curves presenting changes in

the viscosity of the epoxy resins GY-240 and GY-2600 mixed with various amines show a similar trend (Fig. 6). The initial viscosity of the epoxy resins mixed with low-molecular-weight amines EDA and TETA, and phenalkamine Lite-2002 doubles in approx. 25–35 min. The *pot-life* of the epoxy resins mixed with Jeffamine D-230 is more than 120 min. This indicates that the activity of Jeffamine D-230 at room temperature is very low. Nevertheless, this amine could be used as a hardener for epoxy resins at higher temperatures, as it was shown by DSC studies. The shortest *pot-life* (less than 20 min) was characteristic of the systems comprising epoxy resins and phenalkamines NX-6019 and GX-6004. A high reactivity of the phenalkamine GX-6004 in curing epoxy resins was determined also by DSC studies (Fig. 3).

Table 3. **Dynamic viscosity and pot-life of the mixtures of epoxy resins and amine hardeners**

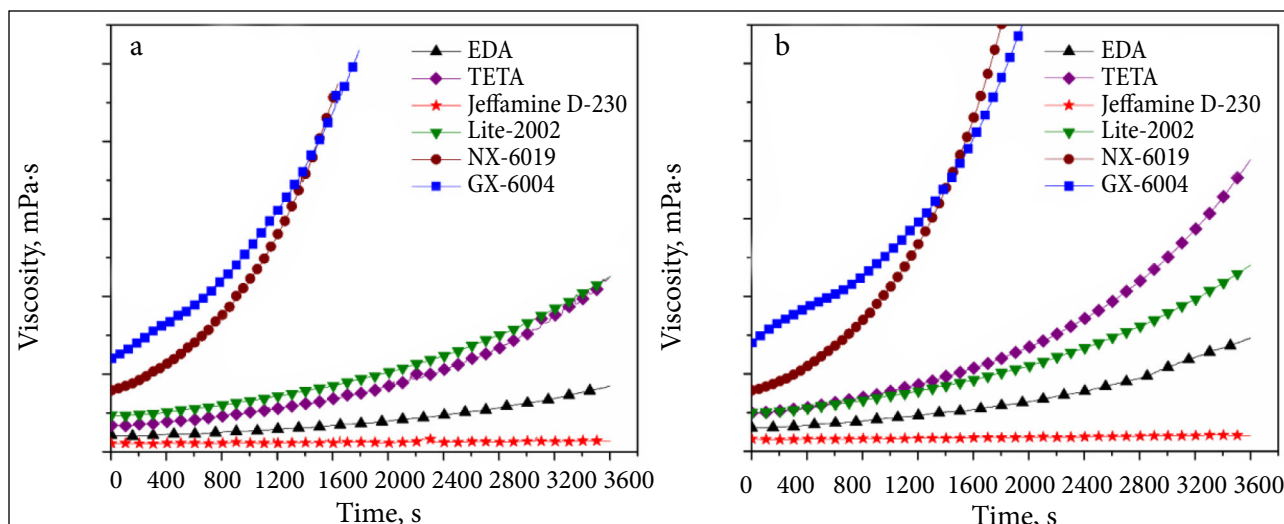| Amine hardener	Initial viscosity, mPa·s	*Pot-life*, min	Initial viscosity, mPa·s	*Pot-life*, min
	Epoxy resin GY-240		Epoxy resin GY-2600	
EDA	1050	35.5	1500	30
TETA	1680	26.5	2490	24.5
Jeffamine D-230	530	>120	790	>120
Lite-2002	2320	30	2510	30
NX-6019	3960	12	3900	12.5
GX-6004	6010	15	7000	19

Fig. 6. Changes in the dynamic viscosity of the epoxy resins GY-240 (a) and GY-2600 (b) after mixing with various amine hardeners

A short *pot-life* of the systems containing epoxy resins and phenalkamine NX-6019 correlates well with the lowest initial curing temperature (at about 48°C) (Table 2), which means that the energetic barrier to start curing reactions is low. Thus, the DSC curing of epoxy resins by phenalkamine NX-6019 starts earlier and, regardless a lower curing rate, reaches full curing in approximately the same time as by using the most reactive phenalkamine GX-6004 (Fig. 2).

CONCLUSIONS

The curing of BPA-based epoxy resins Araldite®GY-2600 and Araldite®GY-240 by low-molecular-weight amines EDA, TETA and Jeffamine D-230, and bio-based phenalkamines Cardolite®NX-6019, Cardolite®Lite-2002 and Cardolite®GX-6004 was studied by DSC and rheology measurements. Usually, the DSC curing of epoxy resins by phenalkamines started at lower temperature, the cure temperature interval was broader, and the curing rates were slightly lower compared to those cured by low-molecular-weight amine hardeners. The enthalpy of the curing by phenalkamines was lower, especially in the case of more viscous epoxy resin GY-2600, which is an advantage. The curing of epoxy resins by phenalkamine NX-6019 was characterised by the lowest initial curing temperature and the shortest *pot-life* while curing by the phenalkamine GX-6004 started later but proceeded with the highest rate. The glass

transition temperature T_g of the cured epoxy resins depended on the type of the hardener and varied from 50 to 98°C. Slightly lower T_g of the epoxy resins cured by phenalkamines is related to the long alkyl chains present in the composition of the phenalkamines, which increases the flexibility and chain mobility in the cured system. Nevertheless, the use of the most reactive phenalkamine GX-6004 provided a possibility to increase T_g of the cured epoxy resins to 80–85°C.

ACKNOWLEDGEMENTS

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EPOKSIDINIŲ DERVŲ KIETINIMO BIOLOGINĖS KILMĖS FENALKAMINAIS IR MAŽAMOLEKULIAIS AMINAIS TYRIMAS DSK METODU

S a n t r a u k a

Dviejų BPA tipo komercinių epoksidinių dervų *Araldite® GY-2600* ir *Araldite® GY-240* kietinimas (tinklinimas), naudojant mažos molekulinės masės aminus EDA, TETA ir *Jeffamine D-230*, taip pat iš kardanolio susintetintus fenalkaminus *Cardolite® NX-6019*, *Cardolite® Lite-2002* bei *Cardolite® GX-6004*, buvo tirtas diferencinės skenuojamosios kalorimetrijos (DSK) ir reologijos metodais. Nustatyti tokie svarbūs parametrai kaip epoksidinių dervų tinklinimo reakcijos entalpija ΔH_R, sutinklinimo laipsnis α ir tinklinimo greitis $d\alpha/dt$, taip pat sutinklintų epoksidinių dervų stiklėjimo temperatūra T_{st}. Naudojant fenalkaminus, tinklinimo reakcijos prasidėjo šiek tiek žemesnėje temperatūroje ir vyko šiek tiek mažesniu greičiu nei kietinimui naudojant mažamolekulius aminus. Tinklinimo fenalkaminais entalpijos pokyčiai buvo mažesni, ypač tinklinant didesnės klampos epoksidinę dervą *GY-2600*. Epoksidinių dervų ir fenalkamino *NX-6019* sistemai buvo būdinga mažiausia pradinė tinklinimo temperatūra ir trumpiausia darbo su kietinama derva trukmė, o tų pačių dervų tinklinimas fenalkaminu *GX-6004* prasidėjo vėliau, tačiau vyko didžiausiu greičiu. Iš kardanolio susintetintų fenalkaminų naudojimas epoksidinėms dervoms kietinti turi privalumų, nes reakcijos pakankamai greitai vyksta net ir kambario temperatūroje, pasiekiamas didelis sutinklinimo laipsnis ir išvengiama kietinamos sistemos perkaitimo. Sutinklintų epoksidinių dervų stiklėjimo temperatūra kito tarp 50 °C ir 98 °C, ir buvo šiek tiek žemesnė, kai tinklinimui buvo naudojami fenalkaminai. Norint stiklėjimo temperatūrą padidinti iki 80–85 °C, kietinimui reikia naudoti reaktingiausią fenalkaminą *GX-6004*.