



A low-temperature fast curing latent catalyst microencapsulated in a porous resin structure



Kazunobu Kamiya^{a,*}, Noboru Suzuki^b

^a FLP Development Section 2, Products Development Department, Optical Solution Products Division, Dexerials Corporation, 18 Satsuki-cho, Kanuma, Tochigi 322-8501, Japan

^b Department of Innovation Systems Engineering, Graduate School of Engineering, Utsunomiya University, 7-1-2, Yoto, Utsunomiya, Tochigi 321-8585, Japan

ARTICLE INFO

Article history:

Accepted 21 April 2016

Available online 26 April 2016

Keywords:

Aluminum complex

Microcapsule

Epoxides

Cure/hardening

ABSTRACT

Low-temperature, fast curing latent catalysts for epoxides were prepared by microencapsulation of aluminum complexes through interfacial and radical polymerization. The resulting resin microcapsules were thermally responsive. Moreover, because the inside of the capsule had a porous structure, a highly active aluminum complex could be forced into the capsule by solvent penetration. The differential scanning calorimetric curve of the encapsulated complex showed an exothermic peak at 100 °C or less. In addition, after a mixture of bisphenol A epoxy resin, the curing catalyst, and triphenyl silanol was stored for 5000 h at 30 °C, the viscosity was only 1.6 times greater than the viscosity just after blending.

© 2016 Elsevier Ltd. All rights reserved.

1. Introduction

Epoxy resin adhesive is easy to handle and has excellent electrical characteristics, and is therefore widely used for electronic part mountings such as multi-layer wiring boards, semiconductor packages, mobile computers, and portable information terminals. In recent years, the reduction in weight and the miniaturization of electronic parts means that the packing density is higher [1]. Adhesives that cure fast at low temperatures are required because excessive heat cannot be applied to the adherend and peripheral components. Furthermore, flame retardant adhesives with lower concentrations of ionic impurities are also desirable.

In 1981, Hayase and coworkers reported a catalyst for initiating epoxy polymerization that was suitable for electronics [2,3]. This catalytic initiator is composed of an aluminum complex and an active silicon compound that forms a Brønsted acid, which induces fast curing of epoxy resin at low temperatures [2–4]. Aluminum complexes are the most suitable for this system because their epoxide polymerization rate is faster than the rate for other organometallic complexes [5]. Moreover, a composite catalyst containing aluminum complexes is appropriate for electronics, which require adhesives to be electrically insulating. The aluminum complex does not function as an ionic impurity in cured resin, unlike conventional Lewis acid cationic initiators [6]. However, because the acid species generated from the aluminum complexes are very active, it is necessary to increase the shelf life of the epoxy resin using a latent initiator.

Murai et al. used phenolic derivatives, which were not active at ambient temperature, in aluminum complex latent initiators in a composite catalyst system. The phenolic moieties generated by the thermal decomposition of the derivatives at 150 °C showed sufficient catalytic activity to initialize epoxy polymerization [7]. Aluminum complexes containing long-chain alkyl groups have also been used, although the solubility in the epoxide was reduced [8]. However, these methods have the drawback of slow curing times. A further latent method is physically separating the catalytic aluminum complex and silicon compound. Microencapsulation of one of the components using interfacial polymerization is a suitable separation method [9–11].

Microcapsules are small spherical containers that are several hundred of nanometers to several hundred micrometers in diameter. They contain solid or liquid materials in an organic or inorganic thin film. Microencapsulation enables sustained release control, powderization of volatile and liquid materials, and the isolation of highly active materials, and has been used in various fields [12–14].

In this work, we have successfully prepared microcapsules containing an aluminum complex by interfacial polymerization of isocyanate. The polymer capsule was a thermally responsive poly (urea-urethane) resin in which the intramolecular hydrogen bonds break when the resin is heated above the glass transition temperature. Breaking the hydrogen bonds reduces the density and increases the permeability of the polymer capsule wall. This allows the epoxy monomer to enter the capsule and come into contact with the catalyst [15]. No aluminum complex derivatives were required for our approach; therefore, the original catalytic activity of the aluminum complex-silicon compound system was retained. We also evaluated the microencapsulated materials as latent epoxy curing catalysts with a view to shelf life extension.

* Corresponding author. Tel.: +81 289 76 0265; fax: +81 289 76 0835.

E-mail address: Kazunobu.Kamiya@dexerials.com (K. Kamiya).

2. Experimental

2.1. Materials

An aqueous solution of the surfactant, sodium *n*-dodecylbenzenesulfonate, SDS (Newrex R, NOF Corp., Japan); dispersion stabilizer, polyvinyl alcohol, PVA (PVA-205, Kuraray Co. Ltd., Japan); the silane coupling agent, 3-glycidoxypropyltrimethoxysilane, 3GPTMS (Silquest A-187 silane, Momentive Performance Materials Japan LLC,

USA); the cycloaliphatic epoxy compound, 3',4'-epoxycyclohexylmethyl 3,4-epoxycyclohexanecarboxylate, EMEC (CEL2021P, Daicel Co., Japan); 1,6-hexanediol diacrylate, HDDA (LIGHT ACRYLATE 1.6HX-A, Kyoisha Chemical Co. Ltd., Japan); trimethylolpropane triacrylate, TMPA (LIGHT ACRYLATE TMP-A, Kyoisha Chemical Co. Ltd., Japan); and glycidyl ether epoxy resin, bisphenol A epoxy resin, BER (EP828, Mitsubishi Chemical Co., Japan) were commercially available and were used without further purification. Divinylbenzene (Merck Co. Ltd., Germany) was washed with 1 N NaOH

Table 1
Chemical structures of aluminum complexes and DI/TMPs.

	$R^1, R^2 = -OC_2H_5, R^3 = -CH_3$	ALC-1
	$R^1, R^2, R^3 = -OC_2H_5$	ALC-2
	$R =$	MDI/TMP
	$R =$	TDI/TMP
	$R =$	XDI/TMP
	$R =$	H6XDI/TMP
	$R =$	(MDI+TDI)/TMP

Trifunctional isocyanate compounds were obtained by reacting 1 mol of trimethylolpropane with 3 mol of diisocyanate compound.

In the formula of trifunctional isocyanate, the substituent R is the moiety of the diisocyanate molecule excluding the isocyanate group.

MDI: methylenediphenyl-4,4'-diisocyanate.

TDI: toluene-2,4-diisocyanate, toluene-2,6-diisocyanate.

XDI: *m*-xylylene diisocyanate.

H6XDI: hexahydro-*m*-xylylene diisocyanate.

(MDI+TDI)/TMP was obtained by reacting 1 mol of trimethylolpropane with 1.5 mol of TDI and MDI.

Download English Version:

<https://daneshyari.com/en/article/7171087>

Download Persian Version:

<https://daneshyari.com/article/7171087>

[Daneshyari.com](https://daneshyari.com)