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Curing kinetics and mechanical properties of epoxy based coatings: The influence of added solvent

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ABSTRACT

Commercial coatings based on epoxy and amine hardener and organic solvent were prepared to investigate the effect of organic solvent on coating properties, their mechanical property and curing kinetics were discussed in terms of different solvent content. The curing degree was characterized by FT-IR, the mechanical properties were studied by tensile and adhesion tests, the non-isothermal curing kinetics and glass transition temperature of epoxy coating were also investigated by differential scanning calorimetry (DSC). The results indicated that the presence of solvent could lower the curing degree thus affecting the cross-linked structure, the tensile strength and modulus of elasticity, while the flexibility was highly improved by adding more solvent into the system. Different adhesive failure modes were observed with the increasing amount of solvent in comparison with that of pure epoxy, it transformed from cohesive to adhesive failure. Furthermore the two parameter autocatalytic kinetic model Šesták- Berggren (*m,n*) was introduced to describe the curing process and it shows a good agreement with the experimental rates. The results also demonstrated that activation energy of curing reaction increased with rising conversion which revealed that curing process was considered to be diffusion controlled.

1. Introduction

Epoxy amine based thermosetting polymers are a very important class of materials that can be used in many industrial fields because of their good mechanical, thermal and chemical properties [1]. However in the preparation process of commercial epoxy resin, there are many human factors that can influence the properties of the obtained epoxy polymer such as the proper dispersion, epoxy/amine ratio, curing temperature [2] and the amount of solvent. Coatings manufacturers use oligomers with epoxy functionality to provide the desired properties of the coating. Many regulators require these coatings to be 100% solids to reduce the VOC emissions. These formulations however can have a relatively high viscosity and therefore the applicator sometimes adds a compatible organic solvent such as xylene into the epoxy system to lower viscosity and improve the paint sprayability and quality of finish [3]. This also helps to promote the efficient mixing of the two components: epoxy resin and amine curing agent. However the solvent can become trapped in the epoxy matrix during the cure process. The solvent may then evaporate from the cured film and this may affect the final properties of the cured system. In this research, we looked at how the loading of solvent affects the cure kinetics and the physical properties of the cured coating.

2. Literature review

In general, relatively few researchers have studied the effect of solvent to the curing behavior and cured properties of epoxy coatings and in the literature few studies have been performed on this effect. It was reported that despite the benefit of better dispersion that solvent offered to the epoxy system, the thermal and mechanical properties could be negatively influenced by the existence of residual solvent in the coating [4]. The encapsulation of solvent in the curing process was regarded as a critical way to change the network topology of the crosslinked epoxy polymer [5]. The glass transition temperature and curing kinetic rate were slightly decreased by adding solvent into the epoxy resin [6]. On the other hand, the adhesive properties, which are directly related to the service lifetime of epoxy coatings have been investigated by many coating researchers [7], but the effect of solvent addition to the adhesion strength has not been studied extensively in the literature. The current research into the curing kinetics of epoxy resins could also provide important information about the curing reaction and even further details about the curing mechanism [8]. By

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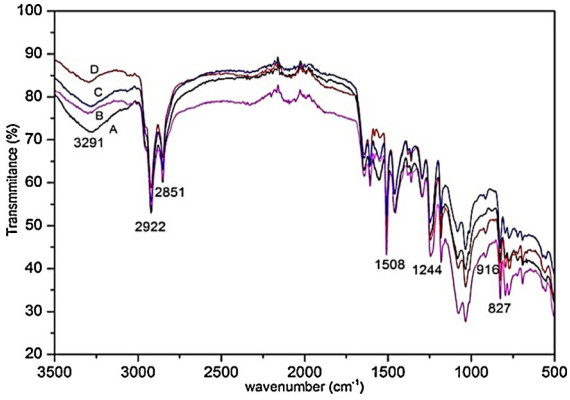

Fig. 1. FT-IR spectra of epoxy coating samples with different solvent content.

Table 1
Sample composition.

Sample	Volume ratio of components Epoxy/amine/solvent (mL)
A	7/7/0.0
B	7/7/0.5
C	7/7/1.0
D	7/7/2.0

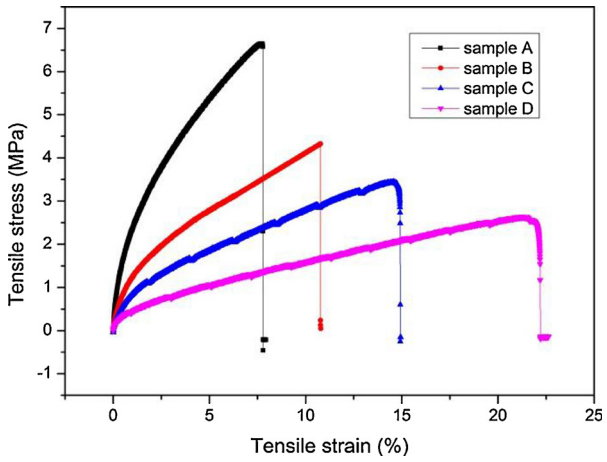

Fig. 2. The tensile stress-strain curve for epoxy coating samples under different solvent content.

Table 2
The tensile parameter values of epoxy coating under different solvent content.

sample	Tensile strength (MPa)	Modulus (MPa)	Elongation (%)
A	6.64	218.08	7.79
B	4.32	112.57	10.77
C	3.43	57.16	14.32
D	2.64	36.35	22.34

introducing suitable kinetic models to describe the curing process, the curing parameters such as reaction order, and activation energy can be obtained.

The goal of this work is to investigate the impact of the organic solvent on the curing reaction as well as the mechanical properties of the resultant coating by employing several characterization methods such as FT-IR, DSC, SEM, tensile and adhesion testing. We selected an appropriate kinetic model to describe the curing rate of the reaction to gain insight into the curing mechanism.

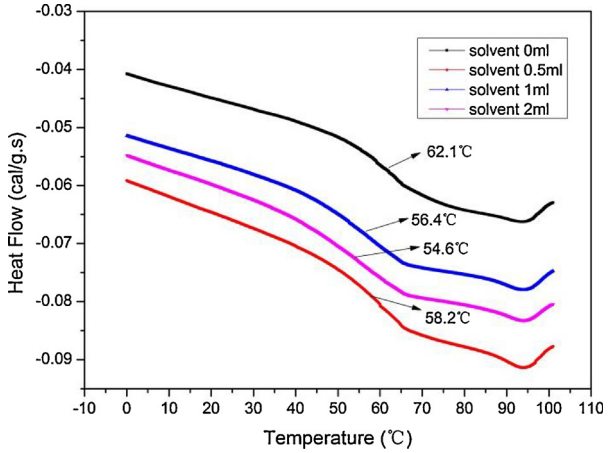

Fig. 3. DSC curves of cured epoxy coatings samples with different solvent content.

3. Experimental

3.1. Materials and preparation

The commercial epoxy polymer matrix used in this study was principally a Diglycidyl Ether Bisphenol A (DGEBA) based epoxy resin, an aromatic amine based curing agent and xylene as a compatible organic solvent all of which were used as supplied by the Berger Paint Company (Dubai). Different epoxy coatings were prepared by adjusting the amount of xylene solvent into the epoxy system. The procedure was as follows: firstly the epoxy and amine hardener were mixed under stoichiometric ratio of 1:1, as per manufacturer's directions, then the solvent was added to the mixture, and stirred until homogeneous. The epoxy resin mix was transferred onto a stainless steel substrate, having previously been cleaned and degreased according to the NACE SP1 surface preparation standard [19]. The coating was spread on the surface using a k-bar with 1.0 mm gap, then the coating was cured at room temperature for 48 h to ensure all solvent had evaporated, but did not perform any long term ageing effect studies. Each sample composition was produced separately 4 times and the average values taken.

3.2. Measurements of mechanical properties

Attenuated Total Internal Reflectance Fourier Transform Infrared spectroscopy (ATR-FTIR) (from Bruker Alpha) was performed in the wavenumber range of 4000–400 cm^{-1} and the curing degree for each sample was determined by comparing the absorption band at 916 cm^{-1} and 828 cm^{-1} which correspond to the stretching vibrations of C—O and C—O—C bonds respectively from the epoxide groups. Tensile testing was performed using an INSTRON tensile tester and was used to measure the mechanical properties; tensile strength, modulus and elongation at break following the testing standard ASTM 638. The adhesion properties were studied by Elcometer 108 pull off adhesion tester. Coatings for adhesion tests were prepared as the same method as mentioned above. The epoxy coating was cured for 48 h at room temperature, and an aluminum dolly glued onto the coating surface with cyanoacrylate adhesive (3M-Scotch) for 30 min to ensure the adhesive had fully attached to the coating. Adhesion strength were recorded after the detachment between coating and substrate. Adhesion tests were carried out three times to check the repeatability of the measurements for all samples.

3.3. Differential scanning calorimetry (DSC) kinetic measurements

The curing kinetics of epoxy coatings were investigated with a DSC Q2000 (TA Instruments) by using the non-isothermal method under