Progress in Organic Coatings 57 (2006) 210–214

PROGRESS IN ORGANIC COATINGS

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Automated determination of pot life of two-component reactive coatings

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Received 24 February 2005; received in revised form 4 November 2005; accepted 11 August 2006

Abstract

The process of pot life screening of two-component coatings is significantly accelerated by using an automated coating formulation system with viscosity measurement capability. High-throughput methods are revolutionizing the processes used in polymer and coatings development. A high-throughput approach to characterize the effect of formulation variables on pot life of two-component solventborne coatings is presented. Using an automated formulation system with viscosity measurement capability, the viscosity of the coatings formulations is measured periodically to determine the viscosity–time profile. As examples, variables such as catalyst type, catalyst level, polyol composition, polyol to isocyanate ratio, and pot life extender were selected as formulation variables and their role in pot life were explored. Compared to traditional pot life determination methods using efflux viscosity cups, the high-throughput approach greatly improves the speed and efficiency of the process and allows many more compositions to be explored.

Keywords: High-throughput methods; Two-component coatings; Pot life

1. Introduction

The high-throughput combinatorial approach – a methodology for creating a number of compounds and then testing them for activity – has been widely adopted in the pharmaceutical industry over the past few years in order to explore wider design space and to understand the effect of different variables in depth. At present, combinatorial and high-throughput methods are finding applications in materials synthesis and analysis [1–6]. Coating formulations with multiple ingredients require a large number of experiments in order to optimize properties. Therefore, the high-throughput method is ideally suited to explore formulations in coating technology [6]. Among the many types of coatings available, polyurethane systems offer high performance in terms of appearance, gloss, durability, hardness and scratch resistance [7]. Two-component polyurethanes are composed of hydroxyl functional polymers, such as acrylics or polyesters, in combination with polyisocyanates. The reaction is catalyzed with organotin compounds and other catalysts. Like epoxy formulations with nitrogen containing curing agent these coatings begin to react immediately upon mixing of the two components and thus have a limited working time, known as the "pot life" [8–10]. Catalyst type, catalyst concentration, pot life extenders, and reactivity and functionality of the reactants can all affect the pot life, making the design of an optimum system challenging. The determination of the pot life involves the tedious repeated measurement of coating viscosity over time, usually using an efflux cup viscometer. This process can be greatly improved through the use of an automated coating formulation system that has viscosity measurement capability. In this study, two different approaches to automated processes for efficiently screening coating compositions for their pot life are considered and illustrated through the use of these approaches in determining the pot life of example formulation libraries.

2. Experimental

2.1. Reagents

Aliphatic polyisocyanate Tolonate HDT90 (HDT) and Tolonate XIDT70B (XIDT) were obtained from Rhodia (Cranbury, NJ). HDT90 is supplied at 90% solids in butyl acetate/

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0300-9440/$ – see front matter
doi:10.1016/j.porgcoat.2006.08.012

aromatic 100 and XIDT 70B is 70% solids in butyl acetate. Tone polyol 0305 (PCL) was obtained from Dow Chemical (Danbury, CT). Dibutyltin dilaurate (DBTDL), dibutyltin diacetate (DBTDA), 2,4-pentanedione, and 1,5-pentanediol, were obtained from Aldrich (Milwaukee, WL). Non-tin catalysts K-KAT XC 6212 (zirconium chelate dissolved in a reactive diluent, Zr Cat A), K-KAT 5218 (aluminum chelate complex, Al Cat), K-KAT 4205 (zirconium metal chelate complex, Zr Cat B) were obtained from King Industries Inc. (Norwalk, CT). Polyurethane grade methyl *n*-amyl ketone (MAK) was supplied by Eastman Chemical (Kingsport, TN). Stock solutions of 1.0 wt.% DBDTL in MAK, 1.0 wt.% DBDTA in MAK and a 90% solution of Tone polyol 0305 in MAK were used to prepare formulations. All the other reagents were used as received.

2.2. Instrumentation

An automated coating formulation system manufactured by Symyx Discovery Tools Inc., was used to prepare the formulations [6]. Materials were dispensed into 24 vials using a robotic pipette having interchangeable tips. Formulations were mixed with a magnetic stir bar that tumbles end-over-end in the bottom of the vial. An automated viscometer mounted on the second robot arm was used to determine the viscosity of the mixtures. The viscometer was calibrated using Brookfield viscosity standards.

3. Results and discussion

The pot life of two-component reactive coatings is an important parameter since it defines the working time of a coating after mixing of the components. Pot life of two-component solventborne reactive coatings may be defined as the time during which viscosity of these coatings remains unchanged [11], the maximum time over which a coating can be applied resulting in constant properties [12], or the time until a noticeable increase in viscosity with the onset of gelation is observed [13]. For this study, pot life was defined as the time required for doubling of the initial viscosity [14]. Maximum allowable time between mixing and application of two-component coatings is guided by their pot life. Working with two-component reactive coatings in an automated coating formulation unit is also a challenge since there may be a time gap between crosslinker addition to the first vial and addition to the last vial. If the pot life is too short, the coating in the first vial may gel before the addition of crosslinker to the other vials is completed, as illustrated in Fig. 1.

Since pot life measurement can be a tedious and time-consuming task, it is logical to seek alternative methods for improving the efficiency of screening coating formulations for their pot life. An automated system that has the capability of dispensing and mixing reagents and measuring the viscosity of the resulting mixture can be used to simplify the experimental process greatly. Conducting multiple measurements in parallel can also significantly increase the number of formulations evaluated in a single experiment.

The Symyx automated coating formulation system was used for this study [6]. The system consists of a two-arm robot. One arm is fitted with an automated pipette with disposable tips and the other arm is fitted with an automated viscometer. The viscometer uses interchangeable measuring tips to prevent cross-contamination of the various samples. Coating formulation ingredients are dispensed into 8 ml vials and mixed using magnetic stir bars.

Two different approaches for the screening of pot life in the automated coating formulation system were evaluated. First, the process of continuous monitoring of viscosity was explored; the procedure is illustrated in Fig. 2. First, all of the non-reactive components were added to each of the vials in the formulation system. Then, the final component – either the crosslinker or catalyst – is added to one vial and the viscosity is measured continuously for that vial until the viscosity has doubled. The process is then repeated for subsequent formulations.

This procedure was evaluated by studying the effect of several different catalysts on pot life. Formulations were based on HDT, PCL, 5% 2,4-pentanedione with the different catalysts. The NCO:OH ratio was 1.1:1.0. All non-tin catalysts were added at their lowest recommended level. The data for these five experiments is shown in Fig. 3. Zr Cat A (zirconium chelate dissolved in a reactive diluent) had the lowest pot life (15 min) and Al Cat (aluminum chelate complex) had the longest pot life (501 min).

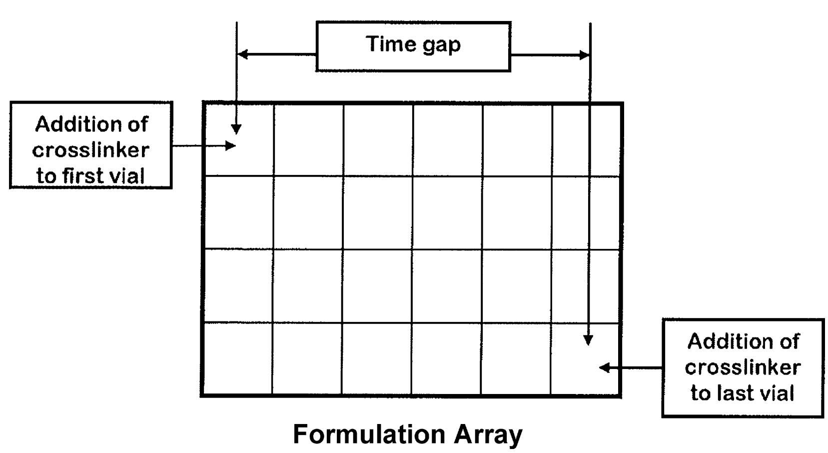

Fig. 1. Illustration of the time lag in dispensing crosslinker from the first vial to the last in the coating formulation system.