Monitoring the formation of polyurethane foams with an infrared camera: Classroom activity

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SUMMARY

Foams prepared with polyurethanes (PU), a polymer with units in the main chain joined by urethane linkages, are commonly used in isolation panels, seals, automative seats, and bedding. The reaction causing the formation of polyurethane foams is a conventional, yet spectacular, way to attract the attention of polymer chemistry students. Infrared cameras are a relatively inexpensive apparatus that allow for semi-quantitatively temperature monitoring at the surface of objects. Because the formation of polyurethane is exothermic, the polymerization reaction and the expansion of the foam can be easily detected by an infrared camera. Information such as volume expansion, maximum temperature, temperature distribution in time and space, and reaction time can be retrieved from short videos. These data can be correlated and discussed in relationship with the reactivity of the components for synthesizing the foam. The primary goal of this activity was to investigate the exothermic reaction of polyurethane foam using an infrared camera which has been successfully used for monitoring the exothermic formation of polyurethane foams. These values can be discussed in term of reactivity of isocyanates (aromatic and aliphatic/cycloaliphatic) for a given formulation of polyols. The highly visual content of such experiments is attractive for children and students. Chemicals for performing the experiments can be donated either individually or as ready sets ("polyols") from the industry. It is anticipated that the investment required for buying the infrared camera can be redeemed by using it for other types of exo- or endothermic reactions and for teaching heat transfer in physics class.

INTRODUCTION

Due to their low density, low thermal conductivity, and mechanical properties, polyurethane (PU) foams have a remarkably broad range of applications including thermal and sound insulation, structural and comfort materials, cushioning, buoyancy, energy absorption (packaging), and aircraft-interior panels (1). PU foams are usually produced by the exothermic addition polymerization between isocyanate groups and polyols that form urethane linkages, called gelling reaction (1). This reaction is carried out at room temperature with catalysts such as tertiary amine and tin compounds (**Figure 1C**). Moreover, water is used as a blowing agent to generate carbon dioxide (CO_2) gas by reacting with molecules containing isocyanate (-N=C=O) groups in a highly exothermic process (**Figure 1D**) (2-3). CO_2 gas forms bubbles inside the reactive mixture, which are stabilized by surfactants (surface active molecules) (4).

The microstructure and morphology of foam depends on many factors, such as the competition between gelling and blowing reactions, mobility of urethane and urea groups, crosslinking density, and specific interactions between polyols and polyurethane segments (5-6). The PU foams are generally classified as rigid, semi-rigid, or flexible, depending on their mechanical properties and core densities (7). Various grades of PU foams can be manufactured by tuning and controlling nature and properties of the starting materials (isocyanates and polyols) (3). The formation of foams is very popular in chemistry teaching classes, despite the toxicity of isocyanates (8), because of the fast and large expansion of foams, which makes the reaction very impressive. Other experiments related to the preparation and measurements of the density of foams were designed for students dealing with their second-year laboratory projects (9). Moreover, polymer foams were produced in at least 1000 secondary schools in Scotland in order to raise the interest of children in chemistry (10). Herein, we combine the preparation of polyurethane foams with another visual detection method to monitor the temperature of the reaction. The advantage of measurements using an infrared camera is that they provide a colourful real-time monitoring of the foaming process while giving quantitative data related to temperatures of the foams.

Generally, temperature is measured by thermometers, thermocouples, thermistors, and resistance temperature detectors that determine the temperature at explicit points and need to be in contact with the measured objects (11). Conversely, infrared thermal imaging (ITI) is a noncontact tool, which maps the temperature distribution of objects with fast response times (12). Thermal imaging technique converts the invisible radiation pattern of an object into visible images. It generates coloured images that represent different temperatures. Originally, ITI was developed for military use, however, over the past two decades, it has become available for non-military purposes (12). ITI can be applied in all applications where temperature differences are required to support diagnosis, evaluation, or analysis of objects. Thus, it has revolutionized the concept of temperature measurement



Figure 1: Chemical structures of polyols (A) and isocyanates (B). Polyurethane foams are formed via a gelling reaction between polyols and diisocyanates to obtain urethane linkages (C) and a blowing reaction between diisocyanates and water to yield urea linkages and carbon dioxide (D).

in a wide range of scientific real time problems in industry (13), agriculture (14-15), and medical applications (16-17).

Recently, ITI was applied to measure the temperature produced in chemical reactions (12,18-19). Infrared camera was also used for teaching heat transfer by conduction, convection, and radiation (20-23). Furthermore, it was introduced as tool for teaching first year undergraduates for monitoring heat of solutions, crystallization, and differences of vapor pressure (24). In this study, students monitored the exothermic formation of PU foams with an infrared camera from different types of isocyanates. Polyurethane foams were manufactured by the reaction of polyol mixture (Figure 1A) with isocyanates in the presence of catalysts (TEA and DBTDL), surfactant (SDS), and blowing agents (DCM and H₂O). The selected isocyanates were either aromatic (tolylene 2,4-diisocyanate, TDI, or polymeric methylene diphenyl diisocyanate, PMDI) or aliphatic/cycloaliphatic (isophorone diisocyanate, IPDI, or tolonate HDT, HDT) isocyanates. We hypothesized that aromatic isocyanates would show a higher reactivity than aliphatic/cycloaliphatic isocyanates (25) due to their aromatic electron withdrawing substituents (26). Thus, the foam produced by aromatic isocyanates would show larger heat generation, which should be detected by the infrared camera. The reactivity of isocyanates should also influence the volume expansion and morphology of the produced foams. We found that the infrared camera can be used to monitor the exothermic formation of polyurethane foams to detect the differences resulting from the isocyanates used. The findings can be used for teaching chemistry to students.

RESULTS

We fixed the amounts of polyols, crosslinkers, surfactants, catalysts, and blowing agents but calculated the amounts of isocyanates so that molar ratios between isocyanate groups and hydroxyl groups were equal (Table 2). Additionally, we used various isocyanates to prepare the PU foams. To observe the evolution of the temperature during the reactions we utilized an infrared camera. Videos recorded with the infrared camera are available in Supporting Information. As shown in Figure 2A, the foam prepared with an aromatic isocyanate such as TDI showed larger heat generation and even caused distortion of the plastic container. Note that the distortion of the cup occurs in a non-reproducible manner and therefore impairs the accuracy of the data extracted from this experiment. Moreover, the foaming process was completed within 19 s with a maximum temperature of 124°C (T_{max}) measured by infrared camera (Table 1). Noticeably, heat generation from foams formed with TDI was larger than heat prepared with PMDI due to the low viscosity and hence larger mobility of TDI and its reaction rate in the mixture. Conversely, we observed a longer reaction time (Table 1) for foams prepared with aliphatic/cycloaliphatic isocyanates (IPDI, HDT, and mixture of HDT and IPDI), suggesting a slower reaction rate compared to aromatic isocyanates. Moreover, the foam prepared with HDT (cycloaliphatic isocyanate) displayed the longest reaction time (~110 s), probably due to the high viscosity of the chemical. After mixing of HDT with IPDI, the reaction time decreased to ~55 s, due to the lower viscosity of the mixture. The dichloromethane:water (DCM:water) ratio was reduced from 8:1 (PU3) to 2:1 (PU6) using IPDI as isocyanate, while the amounts of polyols, surfactant, water (0.1 g) and catalyst were kept constant (Table 1). Interestingly, a decreasing amount of DCM affected the heat generation, rate of reaction, and volume expansion of the foam. Whereas time at T_{max} and time to maximum height increased, the

Entry	lsocyanate	T _{max}	Time at T _{max}	Time to max. height	Volume expansion
		[°C]	[s]	[s]	[fold]
PU1	TDI	124 ± 7	19.0 ± 2.8	2.3 ± 0.5	1.6 ± 0.2
PU2	PMDI	91 ± 5	35.7 ± 9.5	9.7 ± 2.9	2.3 ± 0.1
PU3	IPDI	109 ± 1	71.0 ± 5.7	96.7 ± 2.5	2.9 ± 0.5
PU4	HDT	95 ± 5	110 ± 47	88 ± 26	2.3 ± 0.3
PU5	HDT + IPDI	104 ± 1	55 ± 4	55 ± 3	3.1 ± 0.3
PU6	IPDI	93 ± 10	179 ± 28	189 ± 24	2.5 ± 0.1

Table 1: Maximum temperature T_{max} , time at T_{max} , time to reach maximum height, and volume expansion detected with an infrared camera during the preparation of foams with various isocyanates.



Figure 2: Photographs (A-E) and infrared thermal images (F-J) of PU foams prepared with TDI (A,F), PMDI (B,G), IPDI (C,H), HDT (D,I), and IPDI+HDT (E,J).

volume expansion of foam was reduced when less DCM was used in the formulation (see **Table 1**). As the amount of DCM decreased, the vapor pressure decreased, leading to foams with a lower volume expansion.

We easily visualized temperature gradients via a colour gradient from blue (cold) to white (hot) on infrared thermal images (**Figure 2**). Foams produced from aromatic isocyanates (TDI and PMDI, **Figure 2F-G**) showed less volume expansion than foams produced with aliphatic or cycloaliphatic isocyanates (IPDI and HDT, **Figure 2H-I**). The maximum volume expansion (~3.1-fold) was obtained from mixture of IPDI and HDT (**Table 1**).

Due to its large volume expansion, we selected the foam prepared from mixture of IPDI and HDT to study the effect of reaction time on the reaction temperature. The reaction for producing PU foams can be monitored in time as shown in **Figure 3**. The temperature reached almost 100°C within 1 minute and then decreased until it reached room temperature. The highest temperatures were always located near the bottom of the cup due to the low foaming capability of these formulations.

We also performed scanning electron microscopy (SEM) on cross-sections of the foams to observe the types and sizes of pores produced during the reaction (**Figure 4**). We observed shapeless-cell structures when TDI was used (**Figure 4A**). The structure of foam prepared from HDT showed mostly closed cells (**Figure 4B**), while we detected open cells when the foam was produced by mixture of HDT and IPDI (**Figure 4C**).

DISCUSSION

Foams prepared with aromatic isocyanates such as TDI and methylene diphenyl diisocayante (MDI) makes up 90% of the foams market share. IPDI and HDT are used for producing



Figure 3: Infrared thermal images of PU foams prepared with IPDI+HDT at various time intervals: 1 s (A), 30 s (B), 60 s (C), 120 s (D), 180 s (E), and 328 s (F).

coatings because they provide more UV resistance, abrasion resistance, and weathering resistance (11). Glycerol, a molecule with three hydroxyl groups, acted as crosslinker for providing foams with sufficient rigidity and reduced shrinkage (27). SDS was used for stabilizing a pore structure during foam process. Aliphatic or cycloaliphatic (HDT and IPDI) and aromatic isocyanates (TDI and PMDI) were used because they have different reactivities (Figure 1B). In this work, the polyurethane foam was formed by two main reactions, namely a gelling reaction and a blowing reaction (Figure 1C-D). The polyurethane was formed by the exothermic reaction between hydroxyl -OH groups in the polyol with the -NCO isocyanate groups, resulting in urethane linkages. Additionally, the reaction of isocyanates with water resulted in the formation of urea linkage and carbon dioxide (CO₂), which then acted as blowing agent during foaming process (28). The evaporation of a low boiling solvent such as DCM induced an expansion of foams (29).

In our experimental conditions, the reactivity of isocyanates was the main factor controlling the properties of the foams. The foam produced with TDI showed a larger heat generation than with PMDI. Indeed, TDI is more reactive and displays a lower viscosity than PMDI. Compared to the cycloaliphatic/ aliphatic isocyanates HDT and IPDI, the aromatic isocyanates TDI and PMDI have higher reactivity (low time at T_{max}) because the more electro-positive character of the C atom in NCO groups. Thus, the foams prepared with TDI or PMDI showed larger heat generation, lower volume expansion, and a shapeless-cell structure. Moreover, we found that the foam prepared with a mixture of HDT and IPDI displayed a faster reaction rate than with HDT alone, probably due to the lower viscosity of the mixture. This suggests that the properties of the foams can be therefore tuned by addition of other

Entry	Isocyanate			
	Abbreviation	Туре	Amount (g)	
PU1	TDI	Aromatic	10.8	
PU2	PMDI	Aromatic	16.4	
PU3	IPDI	Aliphatic	13.8	
PU4	HDT	Cycloaliphatic	22.0	
PU5	IPDI + HDT	Aliphatic + cycloaliphatic	7 + 11.4	
PU6	IPDI	Aliphatic	13.8	

Table 2: Types and amounts of isocyanates used in the formulation of the foams. The molar ratio between isocyanate and hydroxyl groups was ~1.

Figure 4: SEM images of PU foams prepared with TDI (A), HDT (B), and IPDI+HDT (C).

isocyanates. Longer reaction time, larger volume expansion, and well-defined morphologies of the foams are caused by a low reaction rate and a high viscosity of isocyanates.

As expected, we detected the highest temperature (white) by the infrared camera in the middle of the foam due to the exothermic nature of the reaction. The foam cells likely elongated quickly and shrunk immediately, resulting from the fast reaction with TDI and the very fast evaporation of DCM blowing agent. It is interesting to note that the heat generated during the reaction was not distributed equally in the foam. This means that the foam prepared in similar experimental conditions are by nature anisotropic, meaning that the properties are different at different locations inside the foams. The students could potentially isolate different portions of the foams and study their density and the morphology and size of the foam cells. The experiments described in the current document were easy to prepare, perform, and interpretate. Quantitative information about the formation of polyurethane foams was retrieved in the form of colourful images. The infrared camera could be further used for monitoring other exothermic reactions in the frame of chemistry classes.

MATERIALS AND METHODS

Safety precautions must be taken prior to and during the experiments. Health effects of isocyanate exposure are occupational asthma, chest tightness, difficult breathing, and irritation of skin, eye, nose, throat, and mucous membranes (26). Isocyanates are classified as potential human carcinogens and known to cause cancer in animals. Dibutyltin dilaurate can cause an allergic skin reaction, damage to organs, severe skin burns, and eye/DNA damage (30). Thus, students are required to wear safety goggles, gloves, mask, and a lab coat during the experiments. The experiment must be set up under a hood.

Materials

Poly(ethylene glycol) 6000 (PEG, 6000 g/mol, Acros Organics), sodium dodecyl sulfate (99% SDS, Acros Organics), tolylene 2,4-diisocyanate (80% TDI, Acros Organics), triethylamine (TEA, Carlo Erba), glycerol (Carlo Erba), dichloromethane (DCM, Honeywell), and dibutyltin dilaurate (95% DBTDL, Sigma) were used as received. Tolonate HDT (HDT) and isophorone diisocyanate (IPDI) were a generous gift from Vencorex, while polymeric methylene diphenyl diisocyanate (PMDI), triethylene glycol (TEG), and bisphenol A (Bis A) were supplied by PTT Global Chemical Company.

Formation of polyurethane foams

The students worked together in pairs. 5 g PEG, 5 g triethylene glycol, and 5 g bisphenol A were mixed with 0.4 g SDS (surfactant) and 0.6 g glycerol (crosslinker) in a plastic cup. The mixture was subsequently incubated at 80°C in an oven for 24 h to be homogenously liquified. In another vial, 0.4 g TEA and 0.5 g DBTDL was mixed with 0.1 g water and 0.8 g dichloromethane (blowing agent). Calculated amounts of isocyanates were added to the mixture of polyols, surfactant, crosslinker, catalyst, and blowing agents (**Table 2**). The mixtures were then stirred for 10 s by vigorous manual stirring.

Observation of foam formation with an infrared camera and foams characteristics

The students observed the temporal evolution of the foam during the reaction with an infrared camera (FLIR T450sc Science/Research Thermal Camera, 60 Hz, w/ 25 Degree Lens, USA). The IR camera was placed in front of a plastic cup at a distance of 32 cm (**Figure 5**). Experimental data such as reaction time, reaction temperature, foam height, and volume expansion could be extracted from the measurements. The students repeated the experiments in triplicate for each formulation. Scanning electron microscopy (SEM, Tabletop Hitachi, 3030Plus) operated at 15 kV was used to observe the cell morphology of PU foams. The foams were placed in liquid nitrogen before being cut prior to SEM imaging.

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Figure 5: Experimental set-up for monitoring foam formation with an infrared camera (left). Volume expansion was estimated by approximating the geometry of the formed foam with a truncated circular cone and by measuring R, h, and r (right). The obtained volume is then $V = 1/3 \pi .h. (R^2+r^2+R.r)$.

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