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A Method for Evaluating the Effect of Blowing Agent Condensation on Sprayed Polyurethane Foams

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RÉSUMÉ

Ce document décrit une technique expérimentale permettant de déterminer l'influence de la température sur la conductivité thermique des isolants en mousse formés par soufflage au moyen de gaz condensables. Cette technique sert à étudier l'effet de l'agent porogène sur la conductivité thermique d'un système polyuréthane formé par soufflage à l'aide de CFC-11 ou de HCFC-123, à divers stades de son vieillissement. On présente aussi une méthode permettant de calculer, à partir des données concernant la conductivité thermique, la pression partielle de l'agent porogène dans le mélange gazeux des échantillons.

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A Method for Evaluating the Effect of Blowing Agent Condensation on Sprayed Polyurethane Foams

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ABSTRACT

An experimental procedure for the determination of temperature dependence of thermal conductivity of foam insulations blown with condensable gases is presented. The procedure is used to investigate the effect of the blowing agent on thermal conductivity of a polyurethane system blown with CFC-11 or HCFC-123, at various stages of its aging. A method to calculate the partial pressure of the blowing agent in the cell gas mixture of the test specimens, from the data on thermal conductivity, is also presented.

INTRODUCTION

Two alternative blowing agents, HCFC-123 and HCFC-141b, have been identified as potential replacements for CFC-11 for manufacturing rigid polyurethane and polyisocyanurate foams. Over the past two years considerable effort has been made to determine physical properties of these new blowing agents and the foams prepared with them [1,2], to enhance a smooth transition from CFC-11 to the alternatives in foam applications. Before the new products are manufactured commercially, more information on the properties of these foams is needed. In an effort to generate such information DuPont Canada Inc. has initiated a series of research projects at the Institute for Research in Construction, NRCC.

The topic selected for initial investigation is the effect of condensable gases on long-term performance of gas-filled cellular plastic (GFCP) insulation. When a GFCP is subjected to a broad range of temperatures, the blowing agent may undergo repeated condensation and evaporation. If this is the case, what is the effect of blowing agent condensation on the thermal performance of the foam? Would

repeated condensation and evaporation of blowing agent affect the polymer structure and diffusion characteristics of the foam? To answer these and similar questions one needs to develop appropriate test methods. These methods should be general enough to permit evaluation of different polymeric materials (polyurethanes, polystyrenes and phenolics) under a broad range of service conditions.

BACKGROUND

Thermal conductivity of a foam insulation that contains a condensable blowing agent, whose normal boiling temperature (T_b) is close to room temperature, varies with temperature, as illustrated in Figure 1. The curve in this Figure can be separated into three regions. In region 1, marked as gas mixture region, thermal conductivity of the foam decreases with temperature. This behavior can be explained using the theory of heat conduction through a mixture of polyatomic gases [3]. The second region, marked as condensation region, is bounded by a minimum and a maximum on the curve. In this region the blowing agent present in the cell gas mixture of the foam undergoes a phase change. At the point *B*, it begins to condense. As the temperature is further decreased, the total amount of blowing agent that is condensed progressively increases and its mole fraction in the cell gas mixture decreases. Since the blowing agent has usually much lower thermal conductivity than any other component such as oxygen or nitrogen, successive reduction in its mole fraction results in a gradual increase in the total thermal conductivity of the cell gas mixture. Hence, the thermal conductivity of the foam increases with decrease in temperature in this region. This increase continues until all of the blowing agent is condensed out of the gas mixture. This happens at the point *C*, which represents completion of condensation of

Figure 1. Temperature dependence of thermal conductivity of a cellular plastic insulation manufactured with a condensable blowing agent.

the blowing agent. In the third region, marked as air-filled region in Figure 1, the thermal conductivity of the foam again decreases with temperature. This decrease, as in the gas mixture region, can once again be explained using the theory of heat conduction through a mixture of polyatomic gases—in this case, still air.

The dependence of thermal conductivity of the foam on temperature in the condensation region is governed by the temperature-saturation vapor pressure relation of the blowing agent. The general relation between temperature and saturated vapor pressure of blowing agents is shown in Figure 2. If the partial pressure of the blowing agent in the cell gas mixture is equal to 1 atmosphere, the condensation will start at T_b . If the partial pressure is below 1 atmosphere, say P_x in Figure 2, the condensation will start at a lower temperature, T_x . Thus information on the point B_c in Figure 1 and temperature dependence of saturation partial pressure of the blowing agent (Figure 2) may lead to the determination of partial pressure of the blowing agent. For blowing agents such as HCFC-123, HCFC-141b or CFC-11, the temperature dependence of saturated vapor pressure may be approximated by Clausius-Clapeyron equation:

Figure 2. Relation between saturated vapour pressure and temperature of blowing agent; T_b is the normal boiling temperature and P_x is the saturated vapour pressure at temperature T_x .

$$\ln(p_o/p) = \frac{H(T_b - T)}{R T_b T} \quad (1)$$

where p is the vapor pressure at temperature T , T_b is the boiling temperature of the blowing agent at standard atmospheric pressure p_o , H is its molar enthalpy of vaporization and R is the universal gas constant. Equation (1), along with the information on the point B_c in Figure 1, can thus be used to calculate the partial pressure of the blowing agent in the cell gas mixture of the foam, knowing the normal boiling temperature and enthalpy of vaporization of the blowing agent.

The objective of the present research project was to develop an experimental procedure that can provide information on the effect of condensation of blowing agents on the thermal conductivity of foam insulations at various stages of their aging. To meet this objective, it was necessary to:

1. Construct and calibrate a heat flow meter (HFM) apparatus that can be used for measurements in the temperature range, 255 to 325 K
2. Develop a technique to "freeze" the aging process of a given specimen, at any desired stage, by preventing inward diffusion of components of air and outward diffusion of blowing agent
3. Develop a test method for determining thermal conductivity of the specimen as a function of temperature, efficiently in terms of time for measurements

This paper presents an experimental procedure that addresses all the three aspects listed above. It also reports experimental data on a sprayed polyurethane foam with two different blowing agents. A foam system equivalent to a commercial spray polyurethane, referred to as "base 88," was selected for this investigation. Two blowing agents were used: CFC-11 and HCFC-123. By selecting the "base 88" system with CFC-11 and HCFC-123, this project provided additional support to a research program initiated jointly by the Institute for Research in Construction, National Research Council of Canada and the Society of Plastics Industry of Canada.

EXPERIMENTAL PROCEDURE

Heat Flow Meter Apparatus

A 300 mm × 300 mm HFM apparatus was built with heat flux transducers [4] on both hot and cold plates. The 300 mm × 300 mm transducer, with 150 mm × 150 mm metering area at the center, was sandwiched between two layers of cork (thickness = 1.5 mm) before it was mounted on the plate. Then a copper sheet of thickness 0.1 mm was glued to the cork to cover the metering area. The remaining part of the cork was covered with Mylar film of the same thickness as the copper sheet. The copper sheet was divided into four squares, each separated by a 0.6 mm gap, and a thermocouple was soldered to the center of each square. The exposed surface of the plate assembly was then painted with black Nextel brand velvet coating, Series 101, to provide an infrared emittance, ϵ of 0.89. The interface of the plate and the cork layer was also provided with four thermocouples. These thermocouples and the ones on the copper sheet can be used to monitor the temperatures equidistant from either surface of the transducer itself and hence to determine the mean temperature of the transducer.

Calibration of the Apparatus

As discussed elsewhere [5], the use of transfer standards developed in a Guarded Hot Plate apparatus of proven accuracy [6] appears to be the most reliable method for calibrating a HFM apparatus.

Two, 300 mm square, expanded polystyrene specimens with thicknesses 25.50 and 25.53 mm and average densities 19.23 and 19.30 kg/m³ were selected for calibration. Their similarity was further checked in the HFM apparatus at fixed hot and cold plate temperatures. The ratios between the electrical output from each heat flow transducer and the temperature difference across the specimens were within 0.04%. This confirmed that their thermal transmission characteristics are identical to the extent required by a pair of transfer standards.

The above pair of specimens was subsequently placed in a 300 mm × 300 mm Guarded Hot Plate apparatus to determine their thermal resistance as a function of mean temperature in the range 240 to 315 K. The results from these tests are plotted in Figure 3. The temperature dependence of the thermal resistance, as shown in Figure 3, is nearly linear for the specimens in the tested range of temperature. This dependence is given by the equation,

$$R = 1.561 - 0.00285 T_m \quad (2)$$

where R is the thermal resistance (m² · K/W) and T_m is the mean temperature (K). Thus, in the range of its validity, Equation (2) can be used to calculate the heat flux across the specimen for any steady state heat transfer condition.

One of these transfer standard specimens was then used to calibrate the new HFM apparatus under steady state conditions. Using ASTM test method C518 and choosing appropriate hot and cold plate temperatures to cover the range of mean specimen temperature of 250 to 325 K, the electrical output from each transducer and thermocouple was measured. The readings from the thermocouples were used to calculate the surface temperature of each plate assembly and the mean temperature of each transducer. Then Equation (2) was used to calculate steady state heat flux across the transfer standard. The electrical output from the transducers and their mean temperatures being

Figure 3. Temperature dependence of the thermal resistance of the transfer standard used for calibration of HFM apparatus.

Figure 4a. Calibration factor of the transducer on the hot plate.

known, the ratio between the steady state heat flux and measured electrical output was calculated at the mean temperature of the transducer. This quantity, expressed as a function of temperature, serves as the calibration factor for the transducer in subsequent application of the equipment for determining thermal conductivities of other test specimens.

The results from the calibration measurements are plotted in Figures 4a and 4b for the transducers on the hot and cold plates respectively. The calibration factor for each transducer varies non-linearly with temperature in the range of interest to this investigation. Least-squares analyses of the calibration data yielded the following empirical equations for the calibration factor of each transducer:

For the hot plate transducer:

$$S = 79.915 - 0.2966 T_m + 0.000451 T_m^2 \quad (3)$$

and for the cold plate transducer,

$$S = 85.539 - 0.3442 T_m + 0.000569 T_m^2 \quad (4)$$

Figure 4b. Calibration factor of the transducer on the cold plate.

where S is the calibration factor ($W \cdot m^{-2} \cdot mV^{-1}$) at mean temperature T_m (K).

Encapsulation of a Test Specimen

It was decided that the best way to "freeze" the diffusion processes was to encapsulate the specimen with an appropriate coating. Several commercially available coatings and membranes were investigated. For polyurethanes the most promising results were obtained with a coating of an epoxy resin (Canus 1000 Epoxy with No. 68 curing agent).

The efficiency of this coating was established by comparing the change in thermal resistance of encapsulated and open-surface, $300 \text{ mm} \times 300 \text{ mm} \times 29 \text{ mm}$, specimens cut from freshly sprayed polyurethane foam. A pair of such specimens was cut and their thermal resistances were measured and found identical. One of the specimens was then coated with the epoxy resin, approximately 0.2 mm thick, on all sides including the edges and was allowed to cure. After two weeks of laboratory exposure both specimens were retested. During that period the uncoated specimen showed 7% reduction in its thermal resistance while the encapsulated specimen showed only 0.6% reduction. After a further ten week period the thermal resistance of the encapsulated specimen was reduced only by 1.2%. This is not a significant change, particularly when a part of this change may be caused by the process of partial pressure equalization due to redistribution of each cell gas component.

Coating with a thin layer (approximately 0.2 mm) of highly conductive material on the specimen surface may increase the lateral component of heat flow and thereby affect the calibration factor S . Even though the calibration of the HFM included some contribution of the lateral flow, a separate test was performed to examine the effect of the epoxy coating on the accuracy of the HFM measurements.

Thermal resistance of an extruded polystyrene specimen of thickness 10.4 mm and density 35.6 kg/m^3 was measured. The specimen, prepared from a sample aged in the laboratory for more than five years, was encapsulated with the same technique used for the polyurethane test series and its thermal resistance was remeasured. This specimen showed a thermal resistance of $0.3375 \text{ m}^2 \cdot \text{K/W}$ before and $0.3292 \text{ m}^2 \cdot \text{K/W}$ after encapsulation. The resistance of the encapsulating layer itself is negligible. Even if the difference of 2.5% is entirely ascribed to the error caused by increased component of the lateral heat flow, for the HFM apparatus described here, this difference is small enough to consider that the measured value of thermal conductivity of the encapsulated specimen is representative of the true value of thermal conductivity.

Test Parameters

Selection of the specimen thickness and the temperature gradient used in this work was based on a compromise between different requirements. The smaller the temperature gradient, the more precise is the determination of the temperature dependence of the thermal conductivity. However, to ensure sufficiently high precision of heat flux measurement one needs a large thermal gradient. Experience with testing 5 to 10 mm thick layers indicated that for the required precision one may use a 10 K temperature difference over 10 mm thick specimens. As discussed

in an earlier publication [7], a period of 1 h was arrived at as the time sufficient to establish steady state and to perform the measurements.

The Test Procedure

The encapsulated test specimen was placed in the HFM apparatus and the hot plate was initially set at approximately 330 K and the cold plate at 320 K. Once the steady state was attained, the electrical outputs from the transducers and all thermocouples were recorded. These values were used to calculate the heat flux at either surface of the specimen, using Equations (3) and (4).

One hour from the start, the temperature of each plate was reduced by 1 K, the new steady state was established and the electrical outputs again recorded. This stepwise decrease in temperature was continued until the hot surface temperature reached approximately 260 K and the cold surface temperature 250 K. Then the measurement process was reversed and the HFM plate temperatures were increased in steps of 1 K. The successive changes in HFM plate temperatures and the data acquisition were managed by a computer. The entire test comprising two measurement series (temperature decrease and increase) takes four to five days.

APPLICATION OF THE TEST METHOD

Sample Preparation

From the core of a given foam sample, a $600 \text{ mm} \times 600 \text{ mm} \times 25 \text{ mm}$ specimen was cut. This specimen was tested immediately for its thermal resistance in a $600 \text{ mm} \times 600 \text{ mm}$ HFM apparatus. This value served as an initial reference value. Then this specimen was further divided into four $300 \text{ mm} \times 300 \text{ mm}$ slabs and from each of these two 10 mm thick specimens were prepared. Four such specimens were then encapsulated at different stages of aging. The other four specimens were used to monitor laboratory aging, (i.e., the change in thermal conductivity versus exposure time). The encapsulated specimen remained in the laboratory at least for a period of 7 days prior to testing, to allow for equalization of partial pressures of all components of the cell-gas.

Results

The details on the specimens tested according to the above procedure are given in Table 1. The results on the polyurethane system blown with CFC-11 are shown in Figure 5 and those on the system blown with HCFC 123 are shown in Figure 6.

Table 1. Period of laboratory exposure prior to encapsulation of the test specimens.

Specimen No.	1	2	3	4
	Exposure Period (days)			
System with CFC 11	9	15	20	25
System with HCFC 123	11	15	25	62

Figure 5. Thermal conductivity of encapsulated specimens of polyurethane foam blown with CFC-11, at different stages of ageing. To avoid crowding, only part of the experimental data is plotted (see Figure 7).

DISCUSSION

Gas-filled cellular plastic insulations gradually lose some of their insulating value during their service life. This phenomenon is referred to as aging. Traditionally it was believed that the outward diffusion of the blowing agent was the main cause for aging. It was only recently, with improved understanding of aging [8], that the role of inward diffusion of air and resulting dilution of the blowing agent became apparent.

The experimental results presented here highlight the significance of air diffusion in the aging process. Figure 5 shows that the change in overall thermal performance of the test specimens of polyurethane system blown with CFC-11 during 25 days was appreciable. Yet, during this period, the beginning of condensation (point B_c in Figure

Figure 6. Thermal conductivity of encapsulated specimens of polyurethane foam blown with HCFC-123, at different stages of ageing. To avoid crowding, only part of the experimental data is plotted (see Figure 8).

1), remained unchanged. One may infer from this that the partial pressure of the blowing agent in the cell gas mixture remained constant over this period. Thus the increase in thermal conductivity may be attributed mainly to dilution of blowing agent due to the inward diffusion of air rather than outward diffusion of the blowing agent. The Figure also shows that the test specimens encapsulated after 20 and 25 days are not too different in terms of aging. One could probably assume that these specimens attained equilibrium with air by 20 days of exposure.

Figure 7 which represents one of the specimens shown in Figure 5 allows one to infer that the point B_c for the particular test specimen corresponds approximately to 279.5 K, as follows. The actual deviation from linearity in the gas mixture region starts approximately at the mean specimen temperature of 284.5 K. At that time the coldest layer in the test specimen is approximately at 279.5 K and it is the condensation occurring in the layer that causes the deviation from linearity. (If the measurements were done with an infinitesimally small temperature gradient across the specimen, a sharp minimum in the curve would occur at 279.5 K.)

As previously discussed, information on point B_c can be used to calculate the partial pressure of the blowing agent in the above test specimen. The normal boiling point of CFC-11 is 296.95 K and its enthalpy of vaporization is 24.77 kJ·mol⁻¹ [9]. Then from Equation (1) the partial pressure can be calculated as 0.53 atmosphere.

As stated earlier, the partial pressure of CFC-11 in all the four test specimens appears to be the same. Since these results relate to 9 to 25 day-old specimens, one may infer that the effective initial pressure of the blowing agent could not have been significantly different from 0.53 atmosphere. The term "effective pressure" is introduced here to differentiate this value from the actual initial pressure of the blowing agent. During the foaming process, the temperature of the foam increases and the pressure of the blowing agent reaches at least 1 atmosphere.

Subsequently, the foam cools down and part of the blowing agent gets adsorbed and dissolved by the polymer matrix. In the tested specimens it appears that the above processes have reduced the pressure of the blowing agent to approximately 0.53 atmosphere.

Figure 7 also shows the results from the cooling and heating parts of the measurement procedure separately. For this specimen containing CFC-11 there is no indication of any hysteresis effect due to condensation and vaporization of the blowing agent.

The experimental data on the system blown with HCFC-123 show slightly different trends. Figure 6 indicates that the test specimens from the system blown with HCFC-123 have already attained equilibrium with air after only 11 days, as the three lower curves are placed close to one another. But the measurements done after 62 days exposure indicate significant reduction in thermal conductivity. A shift in the condensation point is also apparent, indicating a measurable change in the partial pressure of the blowing agent between 25 and 62 days of exposure.

Unlike the specimen in Figure 7, the specimen containing HCFC-123 represented by Figure 8 showed hysteresis effect, probably due to condensation and vaporization, and yielded two separate curves during the heating and cooling parts of the measurement. If this effect is dominant at all stages of aging, more information on adsorption, desorption and dissolution of the blowing agent by the solid matrix of the foam will have to be acquired before the

Figure 7. Experimental results on an encapsulated specimen of the foam blown with CFC-11, during three different series of step changes in temperature.

present data can be analyzed correctly for the partial pressure of the blowing agent.

CONCLUDING REMARKS

This paper presented the development and calibration of a modified HFM apparatus, a method to freeze the aging process of thin slices of polyurethane foam insulation by encapsulation and a test procedure to determine the temperature dependence of thermal conductivity of the encapsulated test specimen in a short period of time.

Measurements were performed on a polyurethane system blown with CFC-11 and HCFC-123. The results very clearly establish the effect of condensation of blowing agents on the thermal conductivity of the foam. The data also permit the determination of partial pressure of the blowing agent in a non-destructive way.

Figure 8. Experimental results on an encapsulated specimen of the foam blown with HCFC-123, during three different series of step changes in temperature.

The non-destructive nature of the procedure presented here can be used to investigate the effects of environmental factors on the long-term thermal performance of the insulations, as the foam is exposed to repeated condensation-evaporation cycles—for example, how freeze-thaw conditions may affect the solubility of the blowing agent in the solid matrix of the foam.

Often it may not be necessary to perform the measurements for as large a temperature range as reported in this work. It appears that information on a temperature range, ± 10 K about the minimum value for the thermal conductivity should give all the details necessary for subsequent analysis for partial pressure of the blowing agent. The present method, when integrated with the traditional measurements of the dependence of thermal conductivity of test specimens on time, will provide unique information for predicting thermal performance of gas-filled cellular plastic insulations [10,11].

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