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Analysis of Material Properties of Aged Rigid Polyurethane Foam

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Abstract

Polyurethane foams are organic materials which can be classified as mechanically rigid or flexible. They are chemically inert, relatively unaffected by the presence of water (cracking, swelling, absorption), and can be manufactured in densities from 3-40 lb/ft^3. In addition, polyurethane foams have exceptional mechanical and thermal properties, which support use of polyurethane foams in the 9977 shipping packages. These packages are designed for shipment of special nuclear material and are approved for storage in the K-area complex (KAC) at Savannah River Site. Through the analysis of conditioned foams, a service life of FR-3700 can be estimated for the storage of the 9977 packages. Thus, considering anticipated temperatures of the 9977 packages in the KAC, FR-3700 was conditioned at 160°F, 185°F, 215°F, 250°F, and 160°F with 50% relative humidity, which remains within the service temperature of -320°F – 250°F of the FR-3700 foam in the 9977 packages. Prior studies have analyzed foams after 7, 20, and 30 months of conditioning with the current study analyzing foams up to 55 months of conditioning. Considering the limited information on the effects of radiation on FR-3700, samples were irradiated with a cobalt-60 source at a high and low dose rate of 90.9 krad/hr and 16.67 krad/hr, respectively, to examine the effects of irradiation and dose rate on thermal properties. It was found that foams conditioned up to 55 months had comparable heat capacity values to FR-3700, with a slight increase due to humidity. Results suggest that fire additives began decomposing at higher conditioning temperatures, reducing the average percent mass loss. Overall, irradiation and humidity had minimal effects on thermal properties of FR-3700.

Introduction

Polyurethane foams are organic materials that can be synthesized using diisocyanates or polyisocyanates with diols or polyols [1]. The foams can be characterized as mechanically rigid or flexible, with open or closed cell composition. Polyurethane foam is chemically inert, relatively unaffected by the presence of water (cracking, swelling, absorption), and can be manufactured in a range of densities from 3-40 lb/ft^3 [2]. The primary means of fire retardation for the foams is through additives; the foams intumesce and form char in the presence of high temperatures which facilitates a considerable increase in volume [2]. This expansion assists in the extinguishment of fire by filling cracks or holes, starving the fire of oxygen while simultaneously creating a thermally insulating barrier in most cases. Modifying the properties of the polyurethane foam can yield a variety of characteristics that can be implemented into common applications such as piping insulation, carpet fibers, and chair cushions. In addition to their versatility, polyurethane foams have exceptional mechanical and thermal properties. These properties support polyurethane foams as an excellent component for the 9977 nuclear shipping packages as shock absorber and fire retardant. These shipping packages are designed for containment of special nuclear material and will be stored in the K-Area Complex (KAC) at the Savannah River Site (SRS) (see Figure 1). Thus, for extended storage of the 9977 nuclear shipping packages (see Figure 2), the material properties of the General Plastics 3716 rigid, closed cell polyurethane foam were evaluated for long term performance by comparing conditioned foam to unaged foam.

Figure 1. 9975 storage in K-Area Complex (KAC) [3]

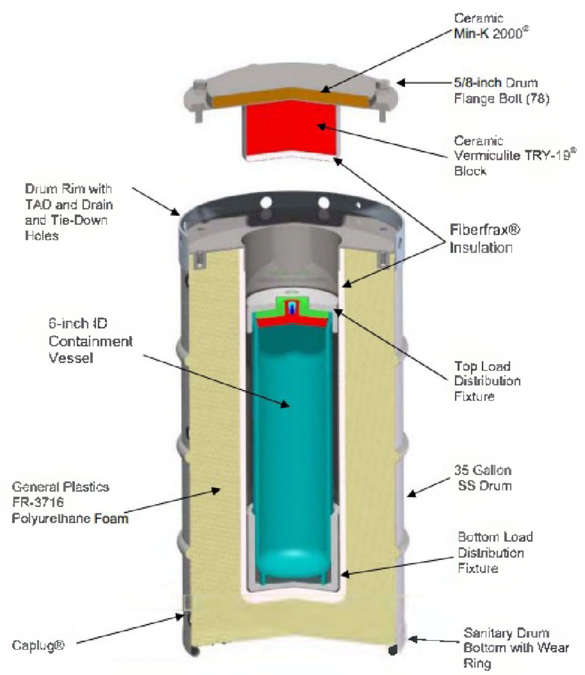

Figure 2. 9977 package diagram [4]

Polyurethane foams can be designed to account for a variety of impact situations, in addition to providing thermal protection [2]. Notably, the primary function of the polyurethane component in the 9977 packages is as a shock absorber, and secondarily as a thermal insulator [5]. Numerous studies have analyzed the mechanical properties of both aged and unaged foams as well as properties of foams that have been irradiated. Few studies analyze the thermal properties of aged and unaged foams, with an absence of research on the effects of radiation on the thermal properties of conditioned foam [2]. The primary factors affecting the performance of the polyurethane foam in the 9977 nuclear shipping packages are moisture, temperature, and oxidation [6]. A 20-year-old piece of FR-3720 (aging conditions were not included in the report) was analyzed and found to have comparable mechanical properties to the pristine sample [2]. This attests to the lifespan of FR-3700, and that the material properties are resilient; however, the conditions of the 9977 package and KAC must be taken into consideration for a reasonable service life estimation. From previous studies, FR-3700 has been proven to be relatively unaffected by water with minimal swelling and water uptake [2], but conditioning temperatures and oxidation affect the material properties of the foam, and consequently, the service lifetime.

The 9977 nuclear shipping packages have the capacity to contain two 3013 containers of nuclear material, which would result in a maximum 38-watt heat loadout (19 watts of heat per 3013 container) [7]. This is an increase in nuclear material compared to the singular 3013 container approved for the 9975 model. If the 9977 package contains the full 38-watt payload, the package can be expected to be near 227°F [7] which approaches the FR-3700 glass transition temperature of 270°F [8]. The typical temperature range in the KAC is between 70-90°F, assuming a conservative average of 85°F, with the maximum temperature recorded in KAC being 104°F [9]. A 38-watt payload with a 100°F KAC temperature could reach temperatures up to 242°F [6]. FR-3700 foams exposed to 250°F for an extended time experienced extreme discoloration and a loss of mechanical properties [6]. More significantly, foams conditioned at 250°F for an extended time did not pass the minimum 50% intumescence requirement for the 9977 packages [6]. Although the 9977 packages have been approved for a 38-watt head load, the KAC limits the packages to a maximum of 24-watt which lowers the maximum temperature of the packages to 173°F with an estimated 85°F KAC temperature [6]. Thus, considering the temperature range that the FR-3700 will be exposed to in the 9977 packages, FR-3700 was conditioned at 160°F, 185°F, 215°F, 250°F in the presence of environmental humidity and 160°F + 50% relative humidity (RH), which remains within the service temperature of -320°F – 250°F for the FR-3700 in the 9977 packages [6].

Another significant capability of the FR-3700 foam is the ability to intumesce. FR-3700 is a thermoset plastic which does not melt, but instead is designed to decompose in the presence of high temperatures in a controlled manner [8]. This expansion is also known as intumescence, which is the primary mechanism of fire retardancy for the FR-3700. The minimum intumescence requirement for the 9977 packages is at least 50% intumescence, but research illustrates that foams at higher conditioning temperatures (250°F) fail to meet this requirement [6]. This signifies that temperature is a substantial component in the degradation of FR-3700 material properties. In addition, prior studies have analyzed the effects of radiation on mechanical properties of polyurethane foam, but there is limited information on the effects of radiation on thermal properties, which could contribute to the degradation of FR-3700 [7]. In addition, a significant deficiency exists examining the effect, if any, of dose rate on thermal properties. A 3013 package in KAC must be able to withstand a systemic 2.2 rad/hr. This dose rate is used for conservatism and is based on the highest dose rate recorded recently at SRS [7]. In the lifetime of a 9977 package, the dose the package would receive can be expected to reach at most 1 Mrad, which is equivalent to a 50-year exposure at 2.2 rad/hr [7]. Considering the limited information of the effects of radiation on the thermal properties of unaged and conditioned FR-3700, samples were irradiated using a cobalt-60 source at a high dose rate of 90.9 krad/hr and low dose rate of 16.67 krad/hr to a cumulative dose of 2 Mrad to examine the effects, if any, of dose rate and irradiation on thermal properties. Prior studies have analyzed foams after 7, 20, and 30 months of conditioning [6,10,11]; the current study analyzes foams up to 55 months of conditioning. Samples were then analyzed for change in material properties by examining physical structure and thermal properties.

Experimental

<u>Environment</u>

Polyurethane foam samples were aged up to 55 months (November 2014 – June 2019) at temperatures and conditions of 160°F, 185°F, 215°F, 250°F, and 160°F + 50% relative humidity (RH). The conditioned samples were then analyzed using Fourier-transform infrared spectrometer (FTIR), a 3D optical microscope, a differential scanning calorimeter (DSC), and a thermogravimetric analyzer (TGA). The foam samples analyzed were small circular aliquots from bulk cubes of aged foam. Samples from the June 2019 collection were irradiated using a cobalt-60 gamma source at a high dose rate of 90.9

krad/hr and low dose rate of 16.67 krad/hr and analyzed via TGA.

Fourier Transform Infrared Spectroscopy

The samples were analyzed via the Jasco FT/IR-6300 Type A which utilized a triglycine sulfate (TGS) detector. The resolution was 2.0 cm^{-1} with 20 collected scans analyzing percent transmittance. A small aliquot of sample was purged in nitrogen gas for at least 45 seconds before data collection.

Optical Microscope

A relatively flat aliquot of sample was placed on the VR-3000 3D optical microscope stage. The samples were imaged before and after the destructive TGA technique for comparison.

Differential Scanning Calorimeter

Aluminum sample and reference pans were chosen in a pair that had approximately the same weight. The target weight for each sample was achieved to be approximately 16 mg. The sample was analyzed via the TA DSC Q20 instrument and with the TA Instrument Universal Analysis 2000 software. The samples purged in nitrogen gas for approximately one minute and then were ramped at 20°C/min to 50°C, held for 10 minutes, ramped at 20°C/min to 150°C, held for 10 minutes, and allowed to cool to room temperature. The resulting data was analyzed via the TA Universal Analysis software.

Thermogravimetric Analyzer

The aluminum sample and reference pan were chosen in a pair with approximately the same weight. The desired weight of alumina is twice the target mass of polymeric samples, thus, approximately 32 mg. The samples were analyzed via the Shimadzu DTG-60 Simultaneous DTA-TG instrument, which utilized building air at a flow rate of +18.15 pccm and had a 0.5 sample scan rate. The instrument began ramping at a rate of 20°C/min to 30°C, held for ten minutes, ramped at a rate of 20°C/min to 550°C, held for ten minutes, and allowed to cool to room temperature. The resulting data was analyzed via the TA60 analysis software.

Irradiator

Samples were irradiated in a cobalt-60 irradiator at a high dose rate of 90.9 krad/hr gamma and a low dose rate of 16.67 krad/hr gamma. Both sets of samples received a total cumulative dose of 2 Mrad gamma.

Results and Discussion

FTIR

All foam samples were analyzed via the Jasco FT/IR-6300 type A instrument which illustrates vibrational energies of molecules. In most of the spectra, significant noise was apparent in the 1750-2500 cm^{-1} range, so analysis of the material properties is not concluded from this region. Figure 3 compares the FTIR spectra from all conditioning temperatures aged for 50 months.

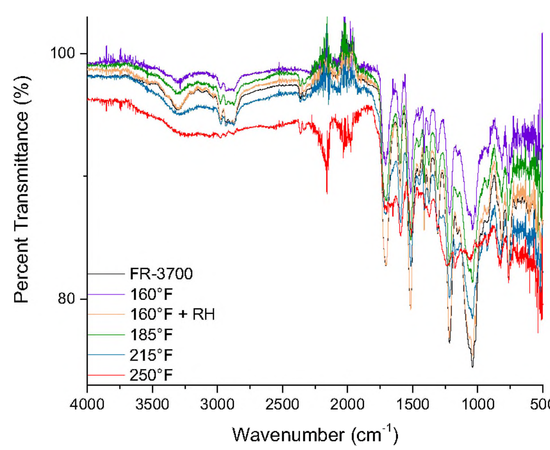

Figure 3. FTIR spectra comparison of all conditioning temperatures aged for 50 months

The peaks are relatively similar between 3500-2750 cm^{-1} and 1750-1000 cm^{-1} for all conditioning temperatures except for the foam conditioned at 250°F, illustrated in Figure 4. The foams conditioned at 250°F have peaks at 3000 cm^{-1} and 3400 cm^{-1} that are smoother and less pronounced than the broad peaks of the FR-3700. Results suggest the beginning of foam degradation, but no significant change to the chemical structure.

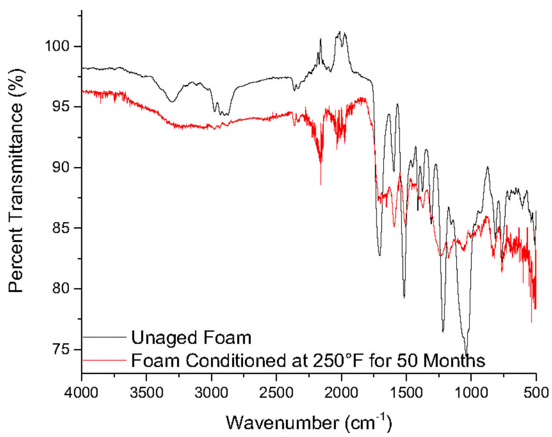

Figure 4. FTIR spectra of FR-3700 and foam conditioned at 250°F for 50 months

3D Optical Microscope

The foams were analyzed via the VR-3000 3D optical microscope. Images were taken before and after the destructive TGA technique, which allows for a comparison of foam structure before and after decomposition. Before the TGA technique, all foams retained the closed cell structure and integrity; after TGA which involves heating the sample to 550°C, the closed cells were degraded, as illustrated in Figure 5. Notably, as the conditioning temperature increased, the discoloration became more pronounced (see Figure 6) while the closed cell structure remained relatively unaffected.

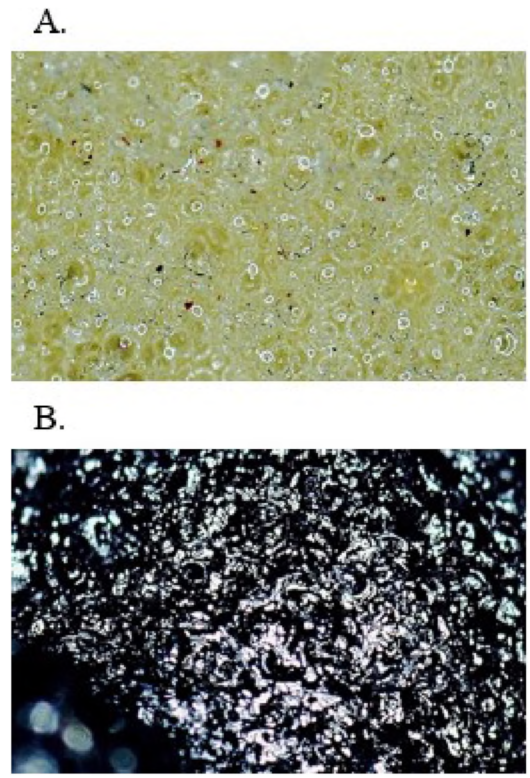

Figure 5. 3D optical microscope images of FR-3700 before (A) and after (B) TGA

Differential Scanning Calorimeter

Due to the long instrumentation collection times and large number of samples, one sample from every calendar year was chosen, ideally the same date for each oven temperature. The heat capacities of the conditioned foam samples were calculated using the heat flow data obtained from the TA DSC Q20 instrument per the ASTM E1269-11 Standard.

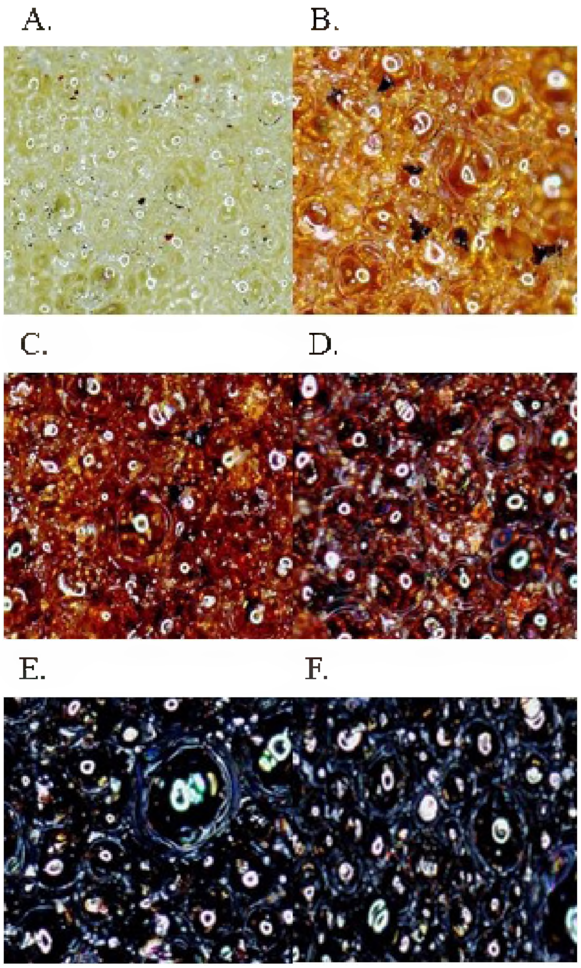

Figure 6. 3D optical microscope images of foams aged 55 months at room temperature (A), 160°F (B), 160°F + 50% RH (C), 185°F (D), 215°F (E), and 250°F (F)

Notably the first heat capacity calculated from the heat flow of the first trial was typically higher than the subsequent trials. This can be attributed to moisture in the sample which was removed by the first trial; subsequent trials had less moisture and lower heat capacity values. Possible conditioning in an environment with a known humidity between trials or collecting a new sample aliquot for each trial would allow for a more representative calculation of heat capacity. The heat capacity was calculated at 71°C and 102°C for all conditioning temperatures, with at least one sample from every year (2014-2019). The specific heat capacity trends at 71°C and 102°C are illustrated in Figure 7.

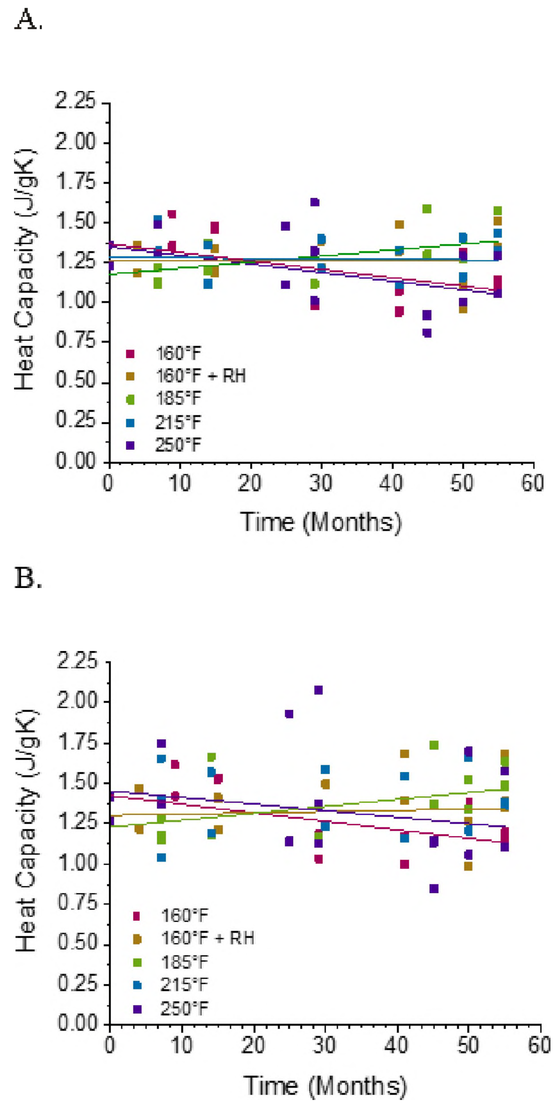

Figure 7. Specific heat capacity at 71°C (A) and 102°C (B) for all conditioning temperatures over time

There was a decrease in specific heat capacity for foams conditioned at 160°C, 160°C + RH, 215°C, and 250°C, with the largest decrease evident with foams conditioned at 250°C. The presence of humidity slightly increased the heat capacity compared to the foams conditioned at 160°F, which signifies a slight benefit from moisture in the sample. In addition, the heat capacity of the foams conditioned at 185°F slightly increased over time. Notably, the trends between the heat capacity at 71°C and 102°C were similar. The trends were not prominent enough to draw defined conclusions; however, the specific heat capacity did not change significantly within conditioning period of 55 months (4.58 years).

Thermogravimetric Analysis

The samples were equilibrated at 50°C before ramping to 550°C to observe the percent weight of the foam during decomposition. The TGA curves for all conditioning temperatures of foams aged 55 months is illustrated in Figure 8.

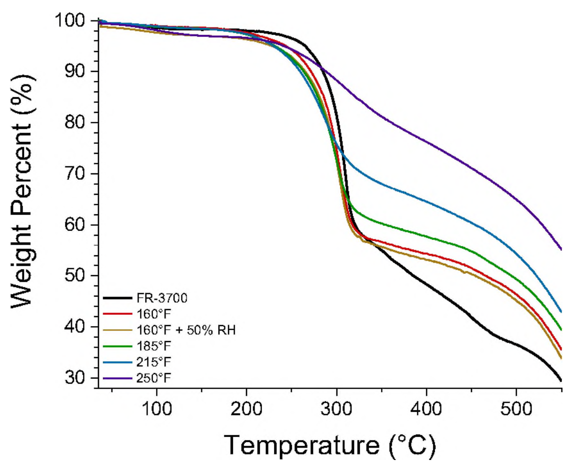

Figure 8. TGA decomposition curves for foams aged 55 months at all conditioning temperatures.

The onset temperature (T_o) and first derivative peak temperature (T_p) can be calculated from the TGA curve. T_o is the point at which weight loss begins. This is found by extrapolation of the initial horizontal plateau of the curve and extrapolation of the linear decrease and calculating the temperature value for intersection of the two lines. This method does introduce considerable uncertainty, especially with the higher conditioning temperatures. The foams conditioned at higher temperatures for longer periods of time had less defined TGA curves which complicated the extrapolation to calculate T_o and first derivative peak temperature. T_p is the temperature at which the greatest rate of change in the mass occurs. This is defined by the largest magnitude decrease in weight percent. T_o and T_p for all conditioning temperatures are illustrated in Figure 9 and 10, respectively.

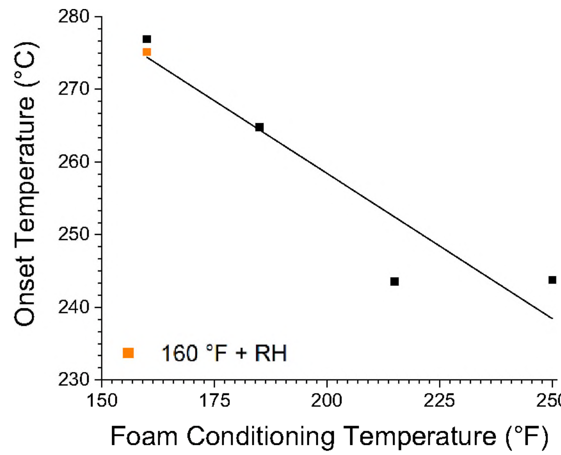

Figure 9. Average onset temperature of all conditioned foams

Another component calculated from the TGA curve is the percent weight loss during the first step of the decomposition reaction. The average percent weight loss is presented in Figure 11, illustrating the change in percent mass loss due to conditioning temperatures.

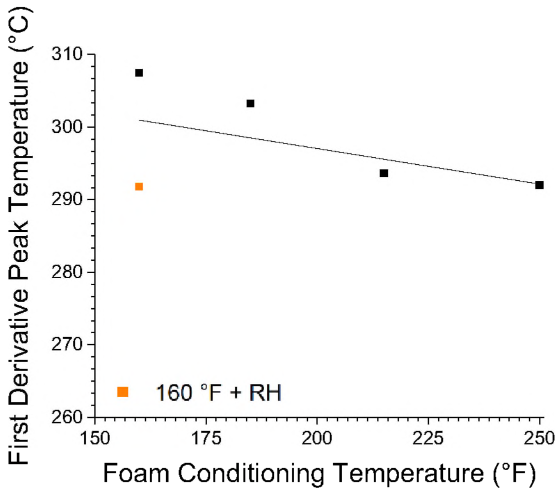

Figure 10. Average first derivative peak temperature of all conditioned foams

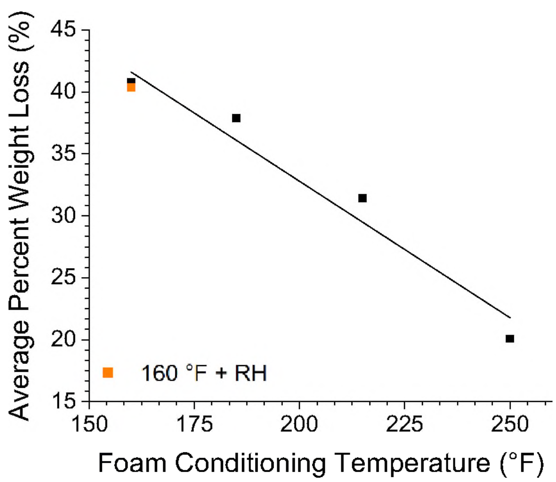

Figure 11. Average percent weight loss of all conditioned foams

The average T_o, T_p, and percent weight loss of all conditioned foams decreased as conditioning temperatures increased. The relative humidity had negliglible effects on the average T_o and percent weight loss, but decreased the average T_p. The decreasing trends signify that as the conditioning temperatures increase, there is a loss in material properties. The decreasing trend in T_o due to conditioning temperatures signifies that the foam mass loss begins at a lower temperature as conditioning temperature increases, in other words, samples aged at higher temperatures are less thermally stable. In addition, the decrease in average T_p signifies that the temperature at which the greatest mass loss occurs begins at a lower temperature due to conditioning temperatures and humidity. Results suggest fire protective additives decomposed at higher temperatures, which contributed to a lower average percent mass loss in higher temperature conditioning.

A valued property of the FR-3700 polyurethane foam is the ability to intumesce. FR-3700 provides fire protection and resistance through the ability to intumesce in exposure to high temperatures and serves as thermal insulation for the 9977 package. To measure the intumescence of conditioned foams, the width of the foam samples was measured with calipers before the TGA experiment. However, the foam samples were extremely fragile and degraded before a final width measurement could be obtained. Although quantitative measurements could not be collected, the foams were inspected visually before and after TGA to evaluate for intumescence. While all foams were examined, only unaged foam and 160°F samples displayed mild intumescence when exposed to elevated temperatures. Notably, this method is through visual inspection only, but would be benefited by standardized intumescent analysis of conditioned foams. The absence of intumescence is most likely due to the degradation of the fire protective additives from prolonged exposure to high conditioning temperatures, illustrated by the decreasing trends in Figures 9-11. This aligns with the TGA decomposition curves of foams exposed to higher conditioning temperatures over time and with the slight changes in the FTIR in Figure 4.

The conditioned foams were not significantly altered in color or size due to irradiation or dose rate. The foams were inspected for intumescence after exposure to high temperatures via TGA analysis. Notably, the irradiated FR-3700, both the high and low dose rate, did not exhibit the intumescence capability of the unaged FR-3700 with no administered dose. The first derivative peak temperature of the low and high dose rate conditioned foams, compared to the foam with no administered dose, is relatively comparable (see Figure 12). The decreasing trend in T_p is similar for all samples. The effect on T_p between high and low dose rate is negligible. The foam conditioned without a dose had a significant effect due to relative humidity; however, the irradiated foams are relatively unaffected due to the presence of humidity.

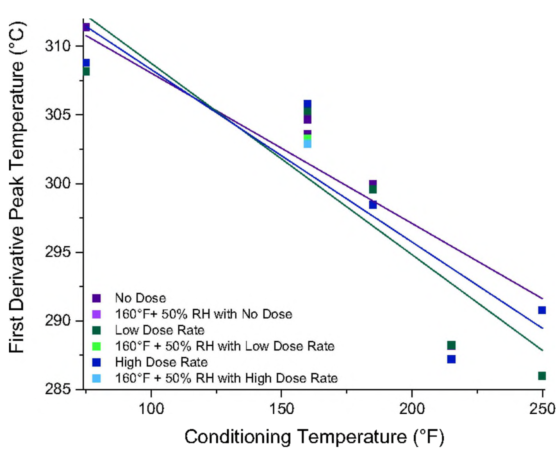

Figure 12. Comparison of first derivative peak temperature for foams conditioned with no dose, a low dose rate (16.67 krad/hr), and a high dose rate (90.9 krad/hr) for 55 months

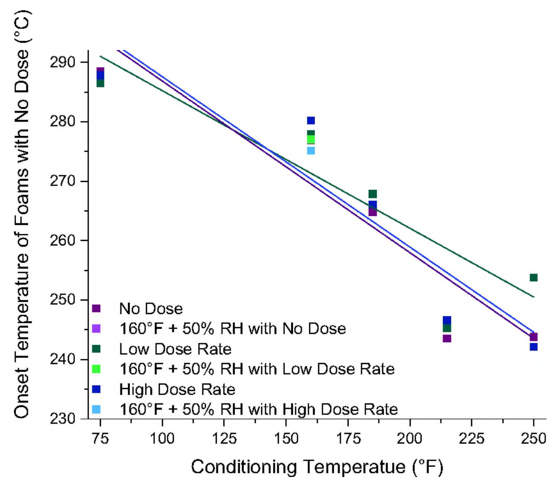

Figure 13. Comparison of onset temperature for foams conditioned with no dose, a low dose rate (16.67 krad/hr), and a high dose rate (90.9 krad/hr) for 55 months

In addition, the onset temperature of the irradiated foam was analyzed (see Figure 13). Foam irradiated were comparable to the T_o of foams without a dose. The effect between high and low dose rate is negligible, as well as any effects due to the presence of humidity.

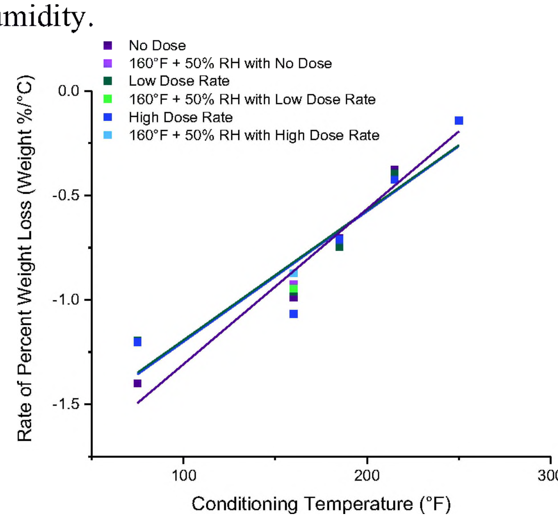

Figure 14. Comparison of rate of percent weight loss for foam conditioned with no dose, a low dose rate (16.67 krad/hr), and a high dose rate (90.9 krad/hr) for 55 months

Lastly, Figure 14 shows the percent weight loss during the first step of the decomposition for irradiated and non-irradiated foam samples. The increasing trend in percent weight loss is similar for all conditioned foams. The trend lines for the low and high dose rate are extremely similar, indicating a negligible effect on percent weight loss due to dose rate. In addition, the irradiated samples and samples that received no dose are comparable, also indicating that the effects on percent weight loss due to irradiation are negligible.

Ultimately, the effects of irradiation on the FR-3700 appear to be minor for the administered cumulative dose of 2 Mrad, which is the expected dose a 9977 package would receive in 100 years of storage in KAC at SRS. The FR-3700 and foam conditioned at 160°F appeared to intumesce less than FR-3700 and foam conditioned at 160°F that received no dose. This indicates that the fire additives responsible for the intumescence capability of the FR-3700 are affected, in some capacity, by gamma radiation. This is significant as intumescence is the primary method of fire retardancy for the 9977 packages. However, the measure of intumescence introduces considerable uncertainty because of the fragile nature of the foams after the TGA analysis; thus, further standardized intumescence testing is necessary for more defined conclusions.

Conclusions

Through microscope imaging, the physical structure of conditioned foam was found to be unchanged. A slight change in chemical structure was found through FTIR analysis. The peaks at 3000 cm^{-1} and 3400 cm^{-1} were smoother and less pronounced for foams conditioned at higher temperatures than for FR-3700. Results suggest fire additives included in foam formulation began to degrade at higher conditioning temperatures.

Heat capacities at 71°C and 102°C, for all samples, were comparable over time. The heat capacities of foams conditioned at 185°F slightly increased over time; in addition, humidity was found to slightly increase heat capacity. The minute changes in heat capacity up to 55 months of aging signify that the FR-3700 has relatively stable heat capacity properties, even at high conditioning temperatures and in the presence of humidity.

Onset temperature and first derivative peak temperature of conditioned foams had similar decreasing trends over time. The average percent weight loss in the first step of the decomposition reaction increased as conditioning temperature increased. In addition, intumescent expansion, after exposure to 550°C, was evaluated and was noticeable for FR-3700 and slightly observed for foams conditioned at 160°F. Conditioned foams aged 55 months, were irradiated at a low and high dose rate, which yielded comparable T_o, T_p, and average percent weight loss values to foams that received no dose. The irradiated foams did not intumesce, regardless of dose rate, to the degree of the foams that did not receive a dose. Ultimately, dose rate and humidity had no significant effect on T_o, percent weight loss values, or intumescent capabilities. While dose rate had no significant effect on T_p, humidity yielded a significant decrease. Irradiation effects on conditioned foam were only evident in the ability of the foam to intumesce. Results suggest that foams at higher conditioning temperatures (215°F and 250°F) experienced a more rapid degradation of thermal properties over time than the foams at lower conditioning temperatures (160°F and 185°F).

The expected conditioning temperature of an average 9977 package with a 24-watt heat loadout in KAC (average temperature 85°F) is 173°F [6]. At this conditioning temperature, FR-3700 would maintain comparable thermal properties to pristine FR-3700 for up to 55 months. Prolonged exposure of the FR-3700 in the 9977 packages to higher conditioning temperatures of 215°F and 250°F is not recommended as results suggest an accelerated decrease of thermal properties.

Future Work

Further thermal degradation testing would be beneficial to evaluate the ability of the conditioned foams to intumesce. A continuation of dose rate effects on conditioned foam would prove beneficial to discern if lower dose rates would alter the effects of irradiation on material properties. Possible conditioning in an environment with a known humidity or analyzing a new aliquot of sample for each trial would provide a more representative heat capacity calculation for the conditioned foams. A continuous evaluation of the conditioned foams to discern more information about the material properties would facilitate a more accurate service life estimation.

Acknowledgements

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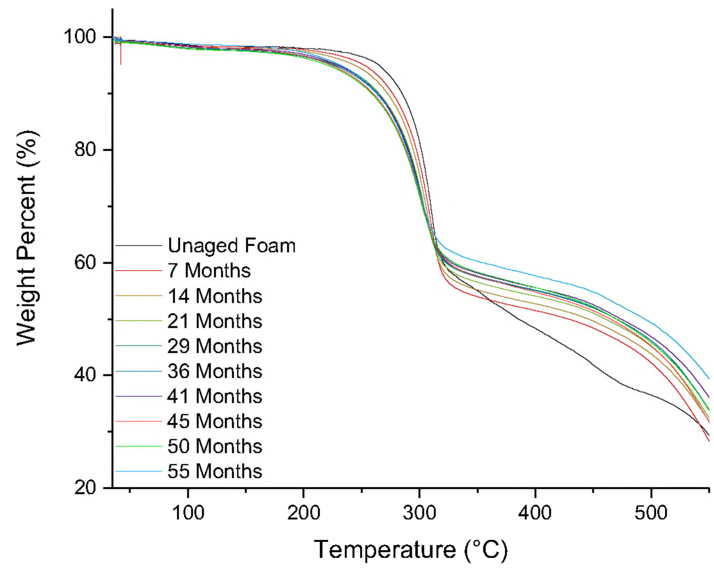
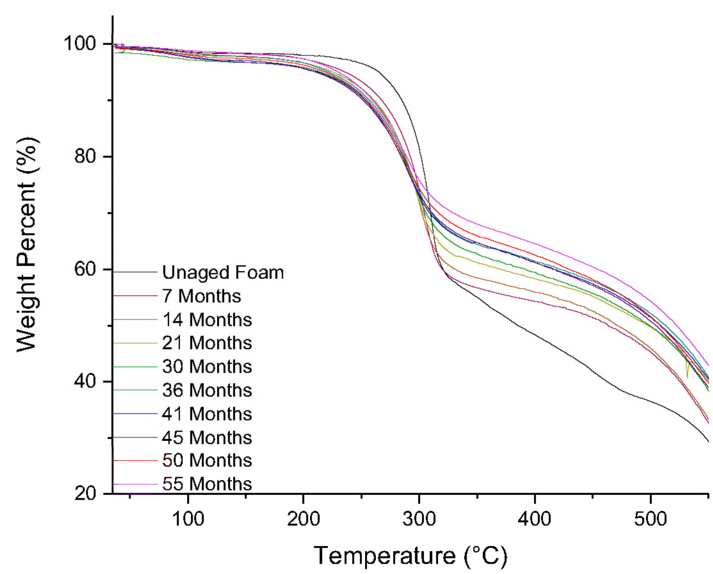
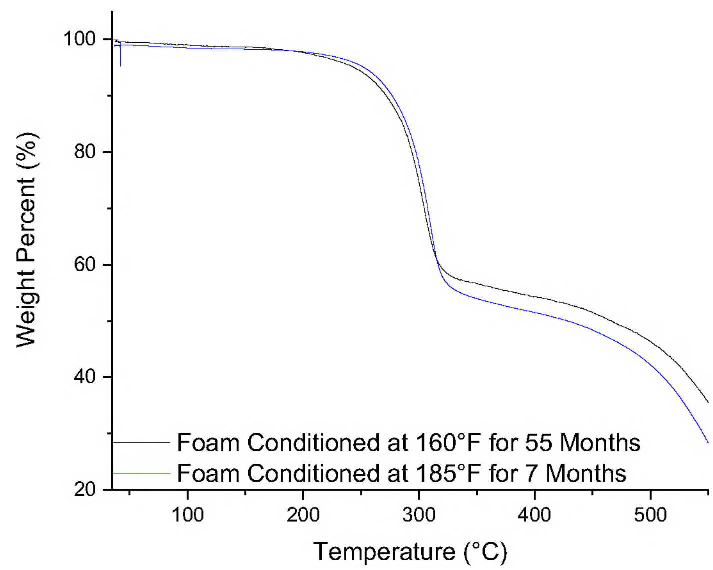
Appendix



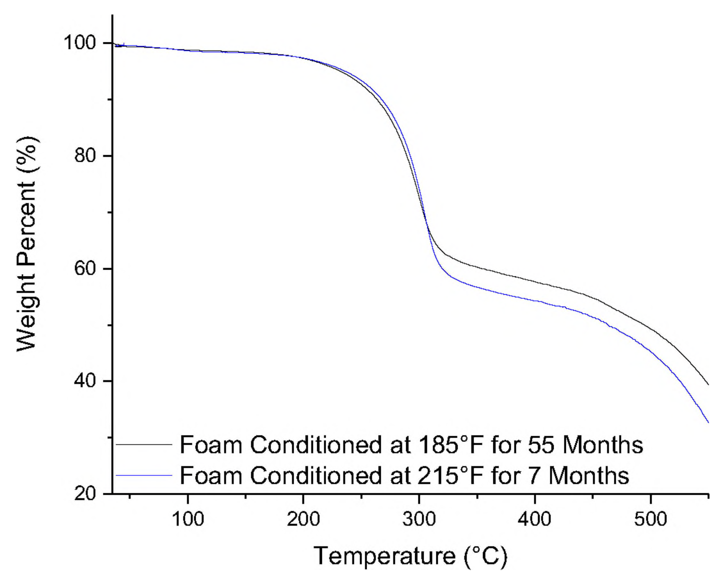
TGA decomposition at a constant conditioning temperature of 185°F over time



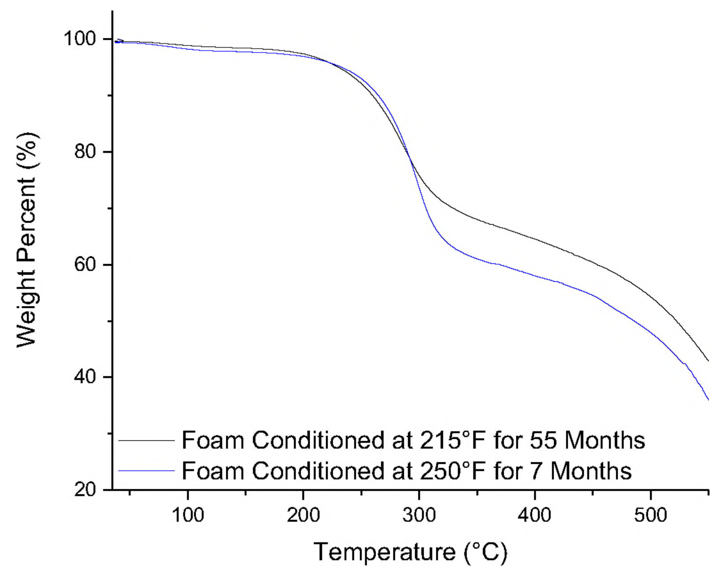
TGA decomposition at constant conditioning temperature of 215°F over time



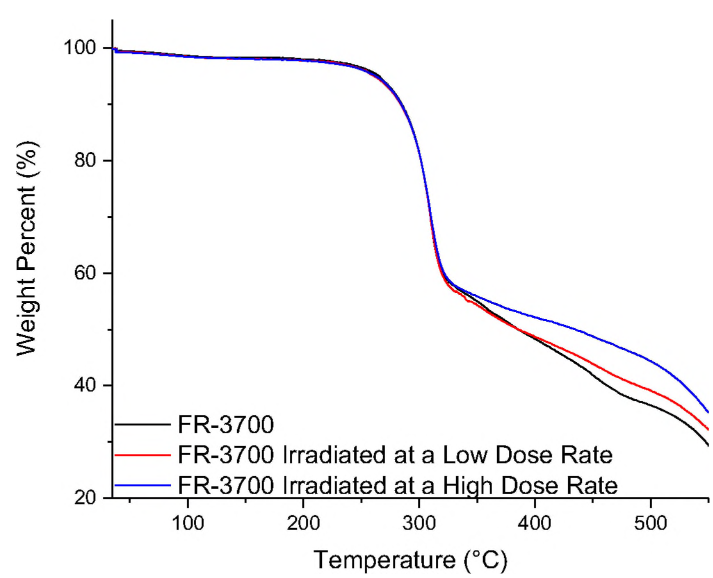
TGA decomposition comparison of foams conditioned longer at a lower temperature to foam conditioned for less time at a higher temperature



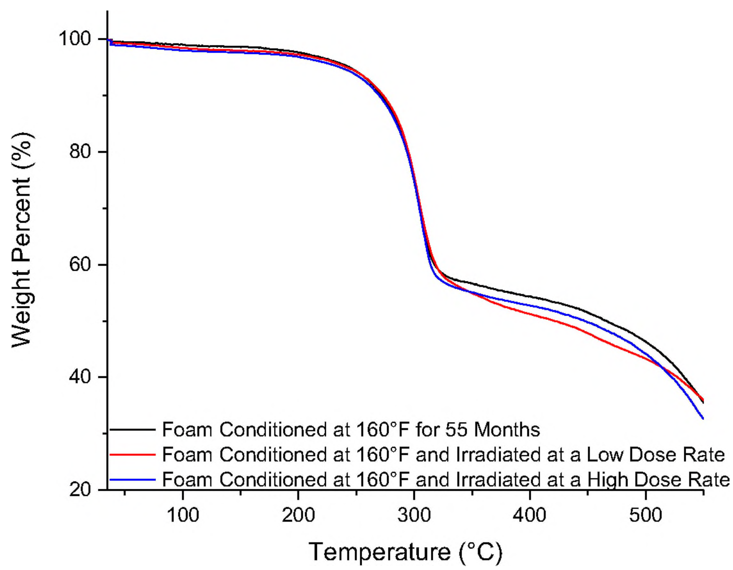
TGA decomposition comparison of foams conditioned longer at a lower temperature to foam conditioned for less time at a higher temperature



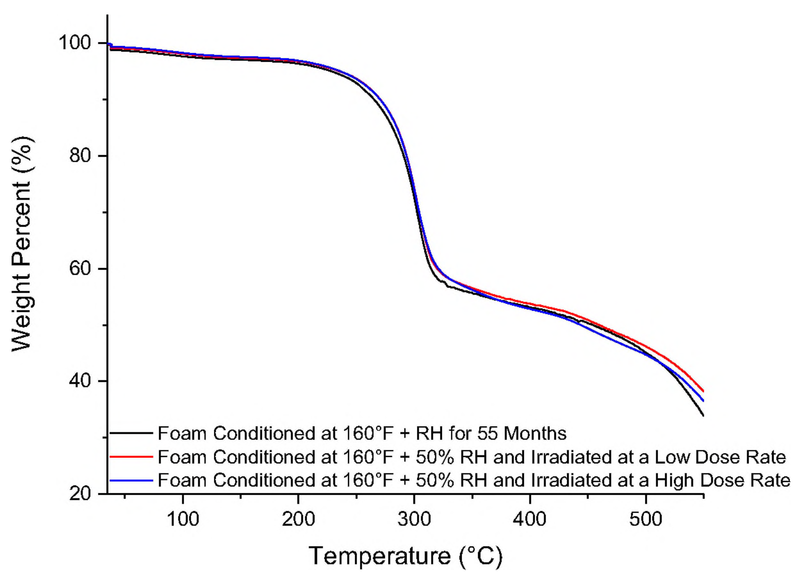
TGA decomposition comparison of foams conditioned longer at a lower temperature to foam conditioned for less time at a higher temperature



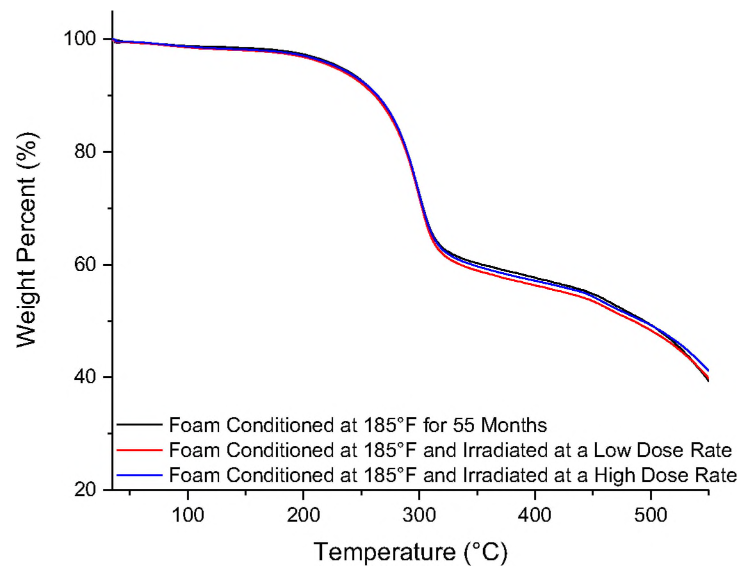
TGA decomposition comparison of unconditioned foam before and after irradiation at a low and high dose rate



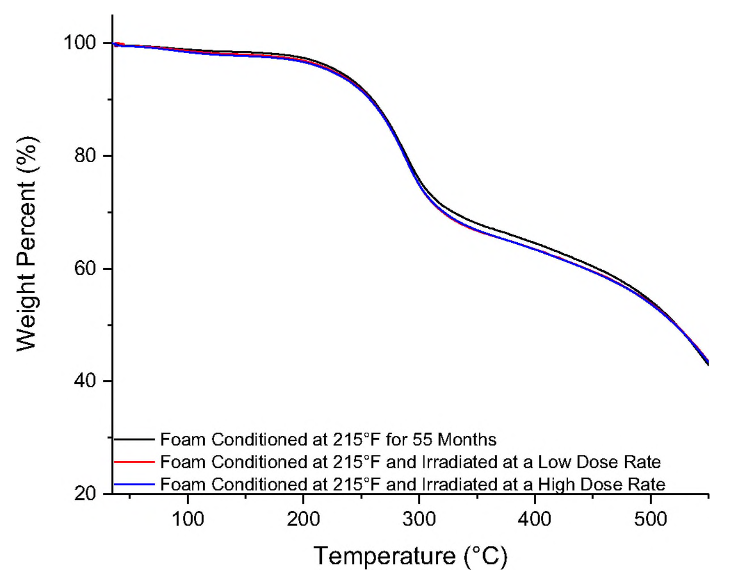
TGA decomposition comparison of foam conditioned at 160°F before and after irradiation at a low and high dose rate



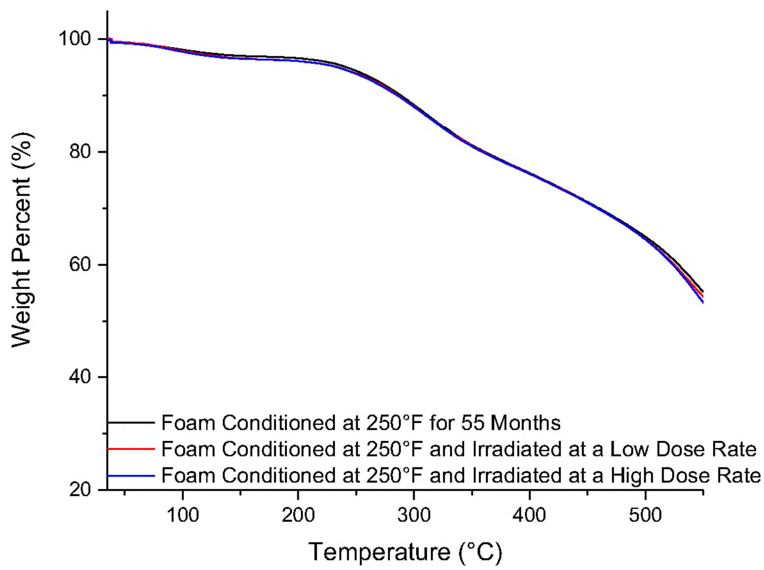
TGA decomposition comparison of foam conditioned at 160°F + 50% relative humidity before and after irradiation at a low and high dose rate



TGA decomposition comparison of foam conditioned at 185°F before and after irradiation at a low and high dose rate



TGA decomposition comparison of foam conditioned at 215°F before and after irradiation at a low and high dose rate



TGA decomposition comparison of foam conditioned at 250°F before and after irradiation at a low and high dose rate