

Compatibility Testing of Polymeric Materials for the Urine Processor Assembly (UPA) of International Space Station (ISS)

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ABSTRACT

In the International Space Station (ISS), astronauts will convert urine into potable water with the UPA by a distillation process. The urine is pre-treated, containing flush water and stabilizers. About 2.5% solids in the urine are concentrated up to 16% "brine" through distillation. Dynamic mechanical analysis (DMA) in the stress relaxation mode was primarily used to test 15 polymeric UPA materials for compatibility with the pre-treated and brine solutions. There were concerns that chromium trioxide (CrO_3), a stabilizer not in the original pre-treat formulation for similar compatibility testing in 2000, could have an adverse effect on these polymers. DMA testing is partially complete for polymeric material samples immersed in the two solutions at room temperature for as long as 200 days. By comparing each material (conditioned and virgin), the stress relaxation modulus (E) was determined for short-term use and predicted for as long as a 10-year use in space. Such a ΔE showed a decrease of as much as 79% for a Nylon material, but an increase as much as 454% for a polysulfone material, with increasing immersion time.

INTRODUCTION

In 2000, UPA compatibility testing was performed on 18 candidate non-metallic materials (17 polymeric, 1 ceramic), and the results were reported (1). Such materials would have different functions on the UPA (e.g. dynamic seal, o-ring seal, drive belt, hose/tubing, bladder, pulley and gear). In 2000, the U.S. pre-treated urine contained primarily sulfuric acid and Oxone (Potassium monopersulfate) as stabilizers. In late 2002, UPA compatibility testing was initiated on 15 candidate non-metallic materials (all polymeric), as several of the materials in the original test matrix were dropped from consideration. This latter testing was performed for a Russian pre-treated urine containing sulfuric acid and chromium trioxide (CrO_3) as stabilizers. Engineers for the UPA at NASA/MSFC expressed concerns that the CrO_3 could have an adverse effect on some of these polymeric materials. In addition, the Russian pre-treat is even more acidic than the U.S. pre-treat, with the former reaching a pH as low as 1.44 for a 13.8% brine solution used in this work.

Data tables in the literature may show the compatibility of polymeric materials with a list of various liquids, but urine is usually not on such lists. However, compatibility of polymeric materials with urine is known to be very important in medical products. Urine bags are often made of flexible polyvinyl chloride or polypropylene (2). The extraction of compounds from human urine has been performed with micro-column liquid chromatography (LC), such as the use of an extraction medium made of fine filaments of Zylon® (a rigid-rod heterocyclic polymer) packed into a capillary tube made of a polymer such as PEEK or PTFE (3).

EXPERIMENTAL

Sample Preparation and Compatibility Testing

Several 1000 ml Nalgene jars with threaded lids were used to contain the solutions and material samples for the compatibility study. For each material tested, four samples were prepared and allowed to hang from a metal rack made from 0.50-in. grid hardware cloth that was immersed in each solution for the duration of the experiment. Grease samples were placed in small metal containers that were also allowed to hang from the test rack. For this testing, the sealed jars were left undisturbed at ambient lab conditions ($\approx 71^{\circ}\text{F}$ / 50% RH) until samples were removed at appropriate times for DMA testing. This study was different from that done previously in 2000, in which the samples were exposed to 100°F / 0.5 psia pressure to more closely simulate actual UPA operating conditions (1).

The polymeric material parts to be used on the UPA are in various geometric shapes. Where possible, flat sheet material was purchased that was the same as or closely similar to the material used to make each part. Flat sheet geometry is more conducive to DMA testing. The Turcon® material was the only one of the 15 candidates not available commercially in sheet form, so material samples were cut from an actual seal part and were kept as flat as possible. For the other materials, samples measuring about 2.5 in. x 0.5 in. x thickness were used for DMA specimens. Thermoplastic and elastomeric materials were mostly used in 0.125-in. thick sheet, as only the thicker Scotch-Brite® pad was used "as is." Rigid thermoplastic samples were prepared in a machine shop, elastomeric samples were cut with a utility knife and straight edge, and Scotch-Brite samples were cut with scissors. With the exception of the grease samples, each material sample was measured for weight and dimension before and after immersion. After each sample was removed from solution, it was rinsed with distilled water, patted dry with a cloth, and measured for weight and dimension. Each material sample was then quickly clamped in the DMA for testing. Scotch-Brite and grease samples were allowed to air dry about 24 hours before any further testing.

DMA Testing

Dynamic mechanical analysis (DMA) in the stress relaxation mode was used as a primary screening test for each conditioned material. Many of these polymeric materials will undergo stress relaxation in real use on the UPA. The TA Instruments 983 DMA was used in this study. Each stress relaxation experiment was performed from 25 to at least 130°C , with an increment of 5°C between stress measurements. An equilibration time of 5 minutes was allowed at each temperature, followed by a displacement time of 15 min. and recovery time of 0 min. For rigid thermoplastic materials, the following DMA sample parameters were generally used: length-to-thickness ratio ≥ 10 ; strain displacement = 0.2 mm; and torque = 10 in.-lbs. For elastomeric materials, the DMA parameters generally used were: length-to-thickness = 2.5; strain displacement = 0.4 mm; and torque = 5 in.-lbs. For Scotch-Brite and Turcon samples, very short sample lengths were used along with a 1.0 mm strain displacement (stress relaxation maximum) to obtain the highest DMA drive signal possible.

By use of TA Instruments time-temperature superposition (TTS) software, the data from each stress relaxation experiment was displayed as a log-log plot of flexural stress relaxation modulus (E) vs. time. The stress data segments were then shifted to short times (lower temperatures) and long times (higher temperatures) to produce a continuous "master curve" over decades of time. For each master curve, the reference temperature chosen within the temperature range measured was 40°C (104°F), which could estimate an average operating temperature of the UPA. In space aboard ISS, the UPA will operate about 8 hours a day. During operation,

polymeric material parts could be exposed to temperatures upwards of 200°F. When the UPA is not in operation, the material parts would be exposed to the ambient cabin temperature. It is also desirable for the UPA polymeric material parts to last a number of years in space. Therefore, TTS software was used to predict E for up to a 10-year use of each material in space. For the stress relaxation testing, a virgin (control) sample kept at ambient lab conditions was tested in addition to the conditioned sample for each material, to yield changes in E due to the urine exposure.

TGA Testing

Two grease samples (Premium RB and Krytox 240 AB) in the material test matrix were screened for changes in weight loss with the TA Instruments 2950 Thermogravimetric Analyzer (TGA). A virgin sample of each grease was tested in addition to samples immersed in the urine solutions. Each TGA heating scan was performed from 30 to 700°C at a linear heating rate of 5°C/min., with an inert gas (helium) sample purge. TGA sample weights were generally 15 mg for Premium RB grease, and 35-55 mg for Krytox 240 AB grease.

RESULTS AND DISCUSSION

DMA and Weight Change Data

Calculations based on DMA stress relaxation data for the UPA thermoplastic and elastomeric materials are summarized in Tables 1 and 2. For the TTS master curve of stress relaxation modulus (E) vs. time generated for each conditioned material sample, a change in modulus (ΔE) was calculated for a short time (t_0) and a long time (t_f). This ΔE was compared as a percentage of the ΔE for the virgin material, as shown in Tables 1 and 2. A value of 100% of the virgin material value for ΔE indicates no net change, which is the most desirable condition. The value of E at t_0 was -3.0 to -5.5 in log time (hours) for most of the materials tested. For most of the thermoplastic materials in Table 1, it was possible to choose E at t_f for log time = 4.9425 hours (10 years) to predict a long-term material performance in space. In Table 2 it was not possible to choose E at t_f for a 10-year prediction for the elastomeric materials, with the exception of Norprene A-55-F (a thermoplastic elastomer). For the other conventional elastomers in Table 2, the stress segments generally did not overlap well, producing a master curve for each material that was usually not very smooth. Because of this, E was determined at t_f for log time = 0.5 hours for these four elastomers.

In Table 1, there were some significant increases in ΔE for the Ultem® 2300 (polyetherimide) and Udel® P-1700 (polysulfone) materials with increasing immersion time, especially for the pre-treated urine solution. This is not of great concern for the Ultem material, since E at t_f for a 10-year prediction is still in the glassy state of the polymer, well above the glass transition temperature (T_g). This is of greater concern for the Udel material, as E at t_f for a 10-year prediction steadily decreased toward/into the rubbery T_g region of the polymer, with increasing immersion time. There is also a significant decrease in ΔE for the Nylatron GSM (a bearing grade of Nylon 66) material with increasing immersion time. This is of great concern because ΔE decreases about 75-80% for both the pre-treated and brine solutions. However, none of the UPA materials stay wetted from the urine solutions, as the UPA operates only about 8 hours/day. A sample of Nylatron GSM immersed in the pre-treated solution for 44 days was tested in the DMA, allowed to dry at room temperature for 2 days, then tested again in the DMA. The re-test yielded a TTS master curve with a ΔE that was 73% of the virgin material value, based on a 10-year prediction.

In Table 2, most of the elastomeric materials showed decreases in ΔE for the pre-treated and brine solutions, with increasing immersion time. It is not uncommon for E at t_f to be about 50% of E at t_0 on the master curve for each elastomer, since each of these materials is well above the T_g at room temperature. However, the EPDM and Neoprene rubber materials appear particularly susceptible, especially to the more acidic brine solution, with a reduction in ΔE of generally more than 40%. In Table 3, the weight increase of the EPDM and Neoprene materials from the brine solution immersion appears to correlate with the DMA data. The weight increase of the Scotch-Brite pad material after conditioning in Table 3 is not of great concern since this is a very porous material.

TGA Data

Table 4 summarizes TGA data on two grease materials: (a) Premium RB, a lithium soap grease for use on bearings; and (b) Krytox 240 AB, a PFPE grease for use on rubber o-ring seals. For temperature onset of major weight loss as the major TGA indicator, the Krytox 240 AB grease was largely unaffected by either the pre-treated or brine solutions. The Premium RB grease was largely unaffected by the pre-treated urine, but the brine solution resulted in an almost 10% loss in thermal stability. However, this change should not adversely affect the long-term performance of the Premium RB grease.

REFERENCES

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2. Axel Bruder et al. *Swiss Plastics*, 1999, No. 4.
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TABLE 1. DMA STRESS RELAXATION DATA ON COMPATIBILITY OF THERMOPLASTIC MATERIALS WITH UPA SOLUTIONS.

Polymeric Material	Immersion time in solution, days (Pre-treat / Brine)	% of virgin material value for $\Delta E (t_0 - t_f)$		E at t_f determined for 10-year prediction? Yes/No
		Pre-treat	Brine	
PTFE Teflon	35 / 36	102.3	93.4	Yes
	80 / 81	103.0	107.1	Yes
	191 / 192	91.1	94.8	Yes
Vespel SP-21 (polyimide)	37 / 38	105.4	114.1	Yes
	82 / 84	105.2	103.5	Yes
	193 / 194	91.2	85.2	Yes
Ultem 2300 (polyetherimide)	37 / 38	219.5	169.9	Yes
	80 / 81	127.4	143.9	Yes
Udel P-1700 (polysulfone)	41 / 42	262.8	79.0	Yes
	84 / 86	454.0	42.5	Yes
Nylatron GSM (Nylon 66)	44 / 45	27.2	25.8	No
	87 / 88	21.3	20.6	No
Turcon (PTFE-based)	47 / 48	105.5	105.2	Yes
	89 / 94	111.3	99.4	Yes
Scotch-Brite pad	49 / 52	201.1	94.7	No
	95 / 97	106.2	131.8	No

TABLE 2. DMA STRESS RELAXATION DATA ON COMPATIBILITY OF ELASTOMERIC MATERIALS WITH UPA SOLUTIONS.

Polymeric Material	Immersion time in solution, days (Pre-treat / Brine)	% of virgin material value for $\Delta E (t_0 - t_f)$		E at t_f determined for 10-year prediction? Yes/No
		Pre-treat	Brine	
Viton V747-75 rubber	39 / 40	82.1	103.3	No
	86 / 87	71.5	89.3	No
	195 / 196	88.3	89.4	No
Norprene A-55-F rubber	42 / 43	88.4	90.5	Yes
	88 / 89	92.9	90.1	Yes
	197 / 199	84.7	82.9	Yes
Butyl rubber	50 / 51	91.6	106.2	No
	90 / 91	72.1	110.6	No
	199 / 200	84.1	99.5	No
EPDM rubber	51 / 52	54.4	58.2	No
	94 / 95	70.6	61.7	No
	201 / 222	58.7	52.7	No
Neoprene rubber	39 / 40	74.4	61.3	No
	82 / 83	63.9	58.6	No

TABLE 3. WEIGHT CHANGES FOR POLYMERIC MATERIAL SAMPLES IMMERSSED IN UPA SOLUTIONS.

Polymeric Material	Immersion time in solution, days (Pre-treat / Brine)	Percent Weight Gain	
		Pre-treated solution	13.8% brine solution
Thermoplastics:			
PTFE Teflon	35 / 36	0.01	0.00
	80 / 81	0.05	0.00
	191 / 192	0.05	0.01
Vespel SP-21 (polyimide)	37 / 38	0.74	0.65
	82 / 84	1.02	0.83
	193 / 194	1.43	1.13
Ultem 2300 (polyetherimide)	37 / 38	0.82	0.62
	80 / 81	0.88	0.63
Udel P-1700 (polysulfone)	41 / 42	0.64	0.54
	84 / 86	0.66	0.55
Nylatron GSM (Nylon 66)	44 / 45	4.56	3.90
	87 / 88	6.14	5.25
Turcon (PTFE-based)	47 / 48	0.49	0.00
	89 / 94	0.65	0.30
Scotch-Brite pad	49 / 52	15.50	0.42
	95 / 97	27.55	30.99
Elastomers:			
Viton V747-75 rubber	39 / 40	0.55	0.46
	86 / 87	0.80	0.75
	195 / 196	1.13	1.32
Norprene A-55-F rubber	42 / 43	0.75	0.83
	88 / 89	1.11	1.17
	197 / 199	1.58	1.61
Butyl rubber	50 / 51	0.48	0.28
	90 / 91	0.64	0.37
	199 / 200	0.98	0.64
EPDM rubber	51 / 52	1.70	4.31
	94 / 95	2.46	5.69
	201 / 222	3.74	7.91
Neoprene rubber	39 / 40	2.38	3.56
	82 / 83	3.07	4.80

TABLE 4. TGA DATA ON COMPATIBILITY OF GREASES WITH UPA SOLUTIONS.

Grease Material	Immersion time in solution, days (Pre-treat / Brine)	% of virgin material value for temperature onset of major weight loss (°C)	
		Pre-treated solution	13.8% brine solution
Premium RB	40 / 40	99.5	96.8
	88 / 88	103.1	91.2
	200 / 200	101.6	90.6
Krytox 240 AB	45 / 45	104.3	103.7
	95 / 96	102.2	102.1