FTIR Analysis Results

Results of the FTIR analysis showed no detectable amount of the contaminants or the solvents remaining on the surface of any of the panels.

Conclusions

All three solvents demonstrated similar effects in each of the natural rubber and EPDM compatibility tests. Plus-4 had the greatest gain in mass with PF Degreaser second and Spirit 126 third.

Although all three solvents were shown to remove the bulk of the natural rubber, only Plus-4 removed all visual traces of the natural rubber from the stainless steel panels. All three solvents removed the EPDM easily with minimal effort. All three solvents removed both the natural rubber and EPDM to levels undetectable by FTIR.

As a result of this testing a preferred cleaner and an alternative were identified. These have been approved and used in the NASA MSFC rubber Lab for over one year now with good results.

Acknowledgements

The authors would like to thank Dink Harris, Jack Simms, Ken Peacock and Troy Daugette in the ODC Lab for their efforts on this project. We also acknowledge the work of Craig Meeks, the Lead Rubber Technician, in his continuing efforts to maintain the rubber equipment in peak condition.

References

Cleaning Ability Test Results

Results of the cleaning ability test showed that Plus-4 removed the natural rubber easily with minimal effort. Both PF Degreaser and Spirit 126 removed the bulk of the natural rubber with moderate effort in less than 1 minute, but both solvents left a stain on the panels. The stain may not be rubber, but some form of oxidation. Complete results can be found in Table III.

Table III: Clean-ability Test Results for Natural Rubber

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Score</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF Degreaser</td>
<td>0</td>
<td>Removed bulk but left stain</td>
</tr>
<tr>
<td>Plus-4</td>
<td>3</td>
<td>Cleaned best</td>
</tr>
<tr>
<td>Spirit 126</td>
<td>0</td>
<td>Removed bulk but left stain</td>
</tr>
</tbody>
</table>

Results of the EPDM clean-ability test showed that all three solvents removed the EPDM easily with minimal effort. Complete results can be found in Table IV.

Table IV: Clean-ability Test Results for EPDM

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Score</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF Degreaser</td>
<td>3</td>
<td>Removed easily</td>
</tr>
<tr>
<td>Plus-4</td>
<td>3</td>
<td>Removed easily</td>
</tr>
<tr>
<td>Spirit 126</td>
<td>3</td>
<td>Removed easily</td>
</tr>
</tbody>
</table>

FTIR Analysis

An FTIR analysis was performed for each contaminant cleaned with each solvent. Toluene was used to liquefy the natural rubber and EPDM. The resultant mixture was then brushed onto stainless steel panels. The panels were allowed to sit at ambient laboratory conditions for 24 hours to allow sufficient solvent flash-off. The contaminated panels were then cleaned with the appropriate solvent and delivered to the FTIR lab for analysis to determine if any residue was left on the panel surface.
Compatibility Test Results

Results of the natural rubber compatibility test showed that Plus-4 had the greatest effect on mass with a change of +56%. PF Degreaser had a change of +38% while Spirit 126 had the least effect with a change of +27%. Complete results for the natural rubber compatibility test can be found in Table I.

Table I: Compatibility Test Results for Cured Natural Rubber

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Mass / 0</th>
<th>Mass / +2</th>
<th>Change</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF Degreaser</td>
<td>6.2018</td>
<td>8.5311</td>
<td>+38%</td>
<td>Swelled / Soft</td>
</tr>
<tr>
<td>Plus-4</td>
<td>6.7630</td>
<td>10.5285</td>
<td>+56%</td>
<td>Swelled / Soft / Sticky</td>
</tr>
<tr>
<td>Spirit 126</td>
<td>6.4396</td>
<td>8.2021</td>
<td>+27%</td>
<td>Swelled / Soft</td>
</tr>
</tbody>
</table>

Plus-4 also had the greatest effect on the EPDM with a change of +36% in mass. PF Degreaser had a change of +25% while Spirit 126 had the least effect with a change of +18%. Complete results for the EPDM compatibility test can be found in Table II.

Table II: Compatibility Test Results for Cured EPDM

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Mass / 0</th>
<th>Mass / +2</th>
<th>Change</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF Degreaser</td>
<td>4.284</td>
<td>5.341</td>
<td>+25%</td>
<td>Light swelling / Sticky</td>
</tr>
<tr>
<td>Plus-4</td>
<td>4.256</td>
<td>5.808</td>
<td>+36%</td>
<td>Light swelling / Sticky / Flaking</td>
</tr>
<tr>
<td>Spirit 126</td>
<td>4.288</td>
<td>5.039</td>
<td>+18%</td>
<td>Light swelling / Sticky</td>
</tr>
</tbody>
</table>

Cleaning Ability Testing

Cleaning ability testing was performed on each contaminant cleaned with each solvent. Stainless steel panels were contaminated with natural rubber or EPDM then cleaned with the appropriate test solvent. To apply the rubber, toluene was used to liquefy the natural rubber and EPDM and the resultant mixture was then brushed onto the stainless steel panels. The panels were allowed to sit at ambient laboratory conditions for 24 hours to allow sufficient time for the solvent to flash-off and for the rubber to be deposited on the panels. The panels were then cleaned with the solvents and rated according to their ability to remove the contaminant from the surface of the stainless steel panels. An explanation of the rating system follows:
Figure 1  NASA MSFC Rubber Mixer

Figure 2  NASA MSFC Rubber Calender Mill
Traditional Cleaning Solvents for Rubber

Traditionally solvents have been divided into 2 classes for cleaning rubber. These are flammable and non-flammable solvents.

Flammable Solvents

The first effective solvents for rubber were the flammable solvents benzene, toluene, xylene, methyl ethyl ketone and hexane. All of these, except hexane, are on the EPA’s list of 17 chemicals targeted for elimination in the workplace due to serious health or environmental risks. Hexane is classified as a volatile organic chemical (VOC) which is a risk for producing low-level photo-chemical smog or ozone. This, in addition to its flammability, makes hexane a poor choice.

Non-Flammable Solvents

The non-flammable solvents were developed and touted as safe replacements for the aforementioned solvents. For years carbon tetrachloride, methyl chloroform, perchloroethylene, and methylene chloride were used with great success. Now these solvents are also targeted for elimination due to uncovered health and environmental risks. Even some of the newer replacements for these chemicals, such as n-propyl bromide, are under a cloud of doubt due to health concerns.

Clearly, in order to comply with newer regulations such as Presidential Executive Orders 12856 and Aerospace NESHAP, all of these older solvents must be considered unsuitable for routine use.

New Cleaning Solvents for Rubber

ATK-Thiokol Propulsion has investigated many replacement solvents and reported the results at previous conferences\(^1\). As a result of this testing three candidate solvents were selected based on their past test performance in attacking rubber. These solvents were each tested for compatibility and effective cleaning ability using both natural rubber and EPDM. An FTIR analysis was also performed for each contaminant cleaned with each solvent.

Compatibility Testing

Compatibility testing was performed on both the natural rubber and EPDM. The purpose of this testing was to determine the solvents ability to dissolve rubber. Samples were first weighed then immersed in the test solvents. After a two-hour immersion time, samples were removed and reweighed to see the effect each solvent had on the mass of each material. Visual observations were also recorded.
Figure 3  Rubber Mixer Cleaning with Natural Rubber

Figure 4  Rubber Mill Cleaning with Natural Rubber
Selection of a Non-ODC Solvent for Rubber Processing Equipment Cleaning

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Abstract

NASA/MSFC has recently acquired new equipment for the manufacture and processing of rubber and rubber containing items that are used in the RSRM system. Work with a previous generation of rubber equipment at MSFC in the 1970's had involved the use of ODC's such as 1,1,1-Trichloroethane or VOC's such as Toluene as the solvents of choice in cleaning the equipment. Neither of these options is practical today. This paper addresses the selection and screening of candidate cleaning solvents that are not only effective, but also meet the new environmental standards.

Background

Rubber and elastomeric compounds play a vital role in the Reusable Solid Rocket Motor. Three kinds of rubber are used for insulation, two for internal insulation of the motor case and one kind for the exterior weather seals and system tunnels. This is in addition to the rubber used in o-rings and other joint thermal protection systems. Because of the number of ingredients and the changing nature of the specialty chemical business, some of the ingredients become obsolete and must be replaced. These changes require new chemicals to be thoroughly screened for their effects on the physical, chemical, and thermal properties of the resulting rubber compounds. Small scale batches of rubber have to be made to screen the new ingredients.

Figure 1 shows the small-scale rubber mixer that has recently been installed at MSFC for the purpose of making and testing small batches of rubber. The mixer is a 6 lb capacity Banbury style mixer that is similar to the full size production capacity mixers. These mixers use tremendous power to literally chew, or masticate, rubber and solid fillers into an acceptable mixture. The mixture is pulled from the mixer and then flattened, or calendered, into a thin sheet with a calender roll mill. Figure 2 shows the calender mill that was recently installed.

Natural rubber is used to clean the mixer and mill by removing gross contamination from the mixer or mill surfaces as shown in Figures 3 and 4. There still remains a film of rubber and traces of raw materials that need to be removed from the mixer or mill. This has usually required the use of solvents.

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