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Abstract
The fiberboard assembly used in 9975 shipping packages as an impact-absorption and insulation component has the capacity to absorb moisture, with an accompanying change to its properties. While package fabrication requirements generally maintain the fiberboard moisture content within manufacturing range, there is the potential during use or storage for atypical handling or storage practices which result in the absorption of additional moisture. In addition to performing a transportation function, the 9975 shipping packages are used as a facility storage system for special nuclear materials at the Savannah River Site. A small number of packages after extended storage have been found to contain elevated moisture levels. Typically, this condition is accompanied by an axial compaction of the bottom fiberboard layers, and the growth of mold.

In addition to potential atypical practices, fiberboard can exchange moisture with the surrounding air, depending on the ambient humidity. Laboratory data have been generated to correlate the equilibrium moisture content of cane fiberboard with the humidity of the surrounding air. These data are compared to measurements taken within shipping packages. With a reasonable measurement of the fiberboard moisture content, an estimate of the fiberboard properties can be made. Over time, elevated moisture levels will negatively impact performance properties, and promote fiberboard mold growth and resultant degradation.

Background
Special nuclear materials (SNM) are stabilized to meet the DOE-3013 Standard and transported in 9975 shipping packages to the Savannah River Site. The SNM remains within the 9975 shipping package, as part of the long-term storage configuration. Technical justification within the storage facility Safety Basis requires surveillance and monitoring of the structural integrity and functional performance of the 9975 shipping packages used to store 3013 containers. As part of the surveillance program, laboratory testing is being performed on small-scale samples to develop service life prediction models for the fiberboard based on material performance under typical and extreme environmental conditions.

The 9975 overpack is fabricated from cane fiberboard (with recent approval of softwood fiberboard as an alternate). The overpack configuration is shown in Figure 1. The fiberboard used to fabricate the overpack must meet specification limits on density and initial moisture content. As the fiberboard layers are laminated with wood glue, both the density and overall moisture content increase. Since fiberboard will readily absorb or
lose moisture based on its surrounding environment (i.e. humidity), further changes in moisture content, and therefore density, are possible throughout the package service life.

The 9975 drum provides a degree of isolation of its contents from the environment, but it does not provide a water-tight seal [1]. The drum contains four ½ inch diameter holes which are normally filled with a plastic caplug. Atmospheric moisture can enter / leave the drum through the bolted closure or around the caplugs, albeit very slowly. Therefore, the moisture level of the fiberboard at the time the drum is closed will continue to influence fiberboard behavior for an extended period. In the event the caplugs become dislodged, the potential for moisture exchange will increase, based on environmental conditions. The fiberboard properties vary with moisture content, and long-term degradation of the material (i.e. loss of strength, reduced thermal conductivity) will occur if the moisture level is sufficiently high.

**Experimental Data: Fiberboard – Moisture Response**

Fiberboard which is not isolated in the 9975 drum will gain or lose moisture so as to approach an equilibrium condition with the surrounding air. Seasonal variations in relative humidity will drive similar cyclic change in fiberboard moisture content, which are manifest as weight change (Figure 2). The humidity which drives the weight change in Figure 2 is consistently high in the summer and early fall, and can vary more widely in the winter and early spring.

Ideally, the moisture content of fiberboard might be measured by weighing the sample before and after exposure to an elevated temperature – the weight change would represent the percentage of moisture (by weight) originally in the sample. However, for large assemblies such as the 9975 overpack, this is not always a convenient or desirable procedure. Most of the moisture content measurements are obtained with a handheld moisture meter, which relates the change in electrical conductivity to changing moisture content. Commonly marketed for applications in solid wood, the meter provides a reading in % wood moisture equivalent (%WME). The conversion from %WME to wt% water varies with wood species and among various cellulosic products. Data were collected on cane fiberboard samples at ambient temperature to correlate the meter output value to wt% moisture. These data are plotted in Figure 3, and indicate the following conversion for cane fiberboard at ambient temperature, over a moisture range of 6 – 40 %WME. Above ~40%WME (~30 wt% moisture) the meter response tends to saturate and does not provide a reliable response.

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\text{Cane Fiberboard Moisture Content (wt\%) = 0.67 \%WME + 2.6} \quad \text{Eq. 1}
\]

Over time, the moisture content of fiberboard will reach equilibrium with the humidity in the surrounding air. Data correlating fiberboard moisture content and relative humidity at room temperature are shown in Figure 4, and present a nonlinear correlation. These data suggest the potential for significant increase in fiberboard moisture content if the ambient humidity approaches 100%. A 9975 package upper fiberboard subassembly was subjected to ~100% humidity for an extended period, and exhibited ~15 % increase in
fiberboard weight (adjusting for the weight of metal within the upper subassembly) due to moisture absorption (Figure 5). This drove the moisture content of the subassembly from ~11 to ~26 wt%. However, the weight that was gained (due to moisture absorption) over ~230 days was lost relatively quickly (within ~90 days) after returning to ambient humidity. After that point, a typical seasonal variation in weight is seen.

As fiberboard absorbs moisture, its properties (e.g. compression strength, thermal conductivity) change. There is an immediate but reversible change in properties as a result of the moisture itself. The moisture may also contribute to or enhance degradation of the fiberboard over time, with additional permanent change resulting from the degradation. Upon absorbing moisture, the fiberboard weight and dimensions will both increase. The overpack will experience the largest dimensional change in its height, perpendicular to the fiberboard layers. The density will also increase, although not as much as the weight [2].

The thermal conductivity and specific heat capacity tend to increase as fiberboard moisture content increases [3]. This change tends to be reversible as the moisture content later decreases [2]. Conversely, the compression strength tends to decrease with increasing moisture content. The area under the stress-strain curve (which is proportional to the energy absorbed by the fiberboard) decreases as the moisture increases. In addition, when tested with the compressive load parallel to the fiberboard layers, the load at which the layers start to buckle also decreases.

Maintaining elevated moisture levels for an extended period can contribute to permanent degradation. The presence of elevated humidity has been shown to increase the rate of fiberboard degradation at temperatures as low as 85°C [4]. While this mechanism will become less pronounced as the temperature decreases toward ambient, the growth of mold becomes increasingly consequential.

**Experimental Data: 9975 Package – Moisture Response**

Small fiberboard samples can provide useful information for understanding the behavior of the material, but performance within a 9975 package might be different, for reasons not immediately obvious. One key consideration with regards to the effects of moisture on the fiberboard is the degree of isolation the drum provides between the fiberboard overpack and the external environment. The larger size of the fiberboard overpack also provides a greater opportunity to develop temperature and moisture gradients, with corresponding variation in fiberboard properties.

Figure 6 shows the behavior of three upper fiberboard assemblies. Initially, each assembly is exposed directly to the room ambient environment, and each experiences a seasonal weight variation of approximately 200g (~2% relative change in weight). After assemblies 1 and 3 were placed in a drum, with the lid loosely in place, the seasonal weight variation dropped to ~25g. Both assemblies initially experienced the same weight variation in spite of one drum being opened weekly while the other was opened much less frequently. This demonstrated that the observed weight changes resulted primarily
from moisture infiltration in/out of the drum rather than from the brief exposures during weighing. Subsequently, the drum containing assembly 1 was bolted shut while the other continued with the lid loosely in place. The seasonal weight variation dropped to ~5g for the assembly in the bolted drum.

There is a potential for the caplugs in the drum to be knocked loose, resulting in an increase in the “openness” of the package. The total area for air infiltration created by removing all four caplugs is ~0.8 sq. inches. In comparison, the leak area of drum 3 (with the loose lid) was estimated by measuring the gap between the drum and lid around the circumference. A measurable gap existed over ~1/3 of the circumference, with a total area of ~1.4 sq. inches. Therefore, the observed seasonal variation of 25g is bounding to that for a properly closed drum with all 4 caplugs missing.

Limited data has been collected to determine whether the correlation between fiberboard moisture and the humidity of the surrounding air (described above) adequately represents the behavior of the overpack inside the 9975 drum. A shallow groove was cut along the side of a fiberboard assembly to permit insertion of a humidity probe, and the assembly returned to its drum. The humidity reading was monitored until it reached a value in equilibrium with the fiberboard moisture content. Humidity readings were taken as the probe was incrementally withdrawn. Moisture readings of the fiberboard were then taken at the same locations after the drum was opened. These data are plotted in Figure 4 along with the lab-scale sample data.

The two data sets show a distinct bias, with the fiberboard moisture in the package being higher than that for the lab samples for the same relative humidity. It is documented that a hysteresis effect occurs in the weight of wood (or fiberboard) as it absorbs or desorbs moisture [5]. However, since the lab sample data set includes both absorption and desorption conditions, this phenomenon does not explain the observed bias. It is more likely that this bias derives from a moisture gradient through the thickness of the overpack.

A moisture gradient will develop in the presence of a thermal gradient, which results from the heat output of the payload. This package had contained a heat load of approximately 9 watts. At the time it was unloaded, a moisture gradient of ~7 %WME was measured between the ID and OD surfaces of the upper fiberboard assembly. The relative humidity data was taken 7 weeks later, and the moisture gradient across the upper fiberboard assembly was 4 %WME (~3 wt%). The moisture gradient was not measured across the lower fiberboard assembly when the package was unloaded, but it was ~5 %WME when the relative humidity data were taken.

Experimental Data: Storage Facility Experience

Up to 6000 packages can be stored in the K-Area Complex (KAC) at the Savannah River Site. Surveillance has been performed on over 160 packages to date. Verification measurements resulted in opening and re-packaging 44 items in alternate shipping
packages, which were examined through the annual maintenance process. Approximately 10 additional drums were also re-packaged in the same manner.

When a package in KAC is opened for surveillance, or prepared for re-use, the axial gap from the top of the drum to the top of the upper fiberboard assembly is measured and compared to a 1 inch maximum criterion. Nine packages from the KAC facility have been identified in which the fiberboard overpack has settled or compacted axially, resulting in an axial gap exceeding 1 inch. Several of these packages were examined in detail.

While the 1 inch maximum criterion was not developed out of concern for excessive fiberboard moisture content, it was observed that each of the packages exceeding this criterion had elevated moisture content relative to a typical 9975 package. In two cases, the amount of excess moisture was significant (estimated at ~2.5 liters), although a specific source of the moisture was not identified. Two general observations were made in examining the 9 packages which exceeded the axial gap criterion:

- The excess moisture present was sufficient to promote the growth of mold on the fiberboard overpack in all but one case. In some packages, only a few small patches of mold were observed. In others, large regions of the outer fiberboard surface were covered with mold.
- The highest moisture concentration was observed in the bottom layers of fiberboard. These layers were compressed, leading to the observed overall decrease in overpack height. The base of the overpack can develop a compressive stress of up to ~3.4 psi from the internal components and payload of the package.

Discussion

The presence of a heat load in a 9975 package creates a thermal gradient through the fiberboard overpack. This in turn causes migration of any moisture existing in the overpack. The moisture concentration will be higher in the cooler regions of the package, which might typically include the top and/or bottom layers. A radial moisture concentration will also develop, with the outer side surface of the overpack containing more moisture than the inner surface.

Any moisture in the top fiberboard layers is effectively trapped under the stainless steel air shield that is attached to the upper fiberboard assembly. However, any moisture that is driven towards the outer surfaces at lower elevations will contribute to an increase in the relative humidity in the annular air space inside the drum. If this increased internal humidity level exceeds the ambient humidity outside the package, it could favor driving some excess moisture out of the drum. However, experience shows that a significant time (e.g. >5 years) can elapse with excess moisture remaining in the package.

Fiberboard properties such as thermal conductivity will vary with moisture content and temperature. As thermal and moisture gradients develop within a package, the thermal conductivity will also change, leading to further change in the thermal and moisture gradients. This level of detail is not addressed in analyses of the 9975 package, but the
The magnitude of its impact is likely small given the margins demonstrated for a conforming package.

The implications of elevated moisture level in the fiberboard overpack vary depending on whether the package is used for transportation or for long-term storage. Both applications rely on the thermal properties of the fiberboard to maintain the product within safe limits. The moisture level impacts both thermal conductivity and specific heat capacity. Elevated moisture also leads to reduced compressive strength, but this is of concern only for storage applications. The fiberboard is not needed for impact absorption in order to demonstrate compliance with transportation accident conditions.

Physical properties are also impacted by the fiberboard moisture level. Increases in both weight and fiberboard height (perpendicular to laminations, axial direction within the 9975 package) are the most significant short-term effects of elevated moisture levels. Lesser impact is seen on radial dimensions and density. When combined with elevated temperature (~85°C and greater), longer-term effects of elevated moisture include a permanent decrease in weight, density and dimensions [4]. At lower temperatures, the growth of mold presents an alternate degradation mechanism.

If removed from the package, the fiberboard assembly would be expected to increase in height as moisture is absorbed, and decrease in height as moisture is lost. However, the weight of the internal components (shield, containment vessels and payload) bears on the lower fiberboard layers within the package. Since increased moisture reduces the fiberboard compressive strength, the lower layers can compact following absorption of moisture, especially when combined with the dynamic loads that might be experienced during transport. In combination with an internal heat load, the primary impact of elevated moisture appears to be a migration of much of the excess moisture towards the bottom layers and compaction of those layers. Relatively little change is seen in height along the sides of the fiberboard assembly. This scenario is consistent with observations on packages removed from storage which did not meet the 1 inch maximum axial gap criterion.

Performing the SARP loading preparation verification of the axial gap ensures excessive fiberboard compression hasn’t occurred. The 1 year certification period for transportation limits the potential degradation that might be related to excess moisture and compacted fiberboard. For packages used as part of a long-term storage configuration, potential degradation modes related to excess moisture have greater potential to advance to a state that compromises package functional requirements. A surveillance program is in place at the Savannah River Site to monitor and predict long-term degradation in storage through package examinations and accelerated aging of laboratory samples.

Conclusions

Migration of moisture into and out of the fiberboard overpack of the 9975 shipping package can occur based on the ambient humidity. However, the drum provides partial
isolation from the external environment, and moisture that is initially within the fiberboard will tend to remain for an extended period. Elevated moisture can have several impacts on fiberboard properties. Short-term impacts of elevated moisture include increased thermal conductivity, decreased compressive strength and increased weight and height. These impacts are reversible as the moisture level decreases. Longer-term permanent impacts of elevated moisture can include decrease in thermal conductivity, further decrease in strength, and the potential for mold growth. The pre-loading verification of the axial gap above the fiberboard provides confidence that significant impacts from excess moisture have not occurred.

References

Figure 1. Cross section of the 9975 shipping package showing the configuration of the fiberboard overpack. The upper and lower fiberboard assemblies are indicated by arrows.

Figure 2. Seasonal variation in weight of small fiberboard sample (~5 cm cube) maintained at ambient conditions. Weight variation is driven by seasonal humidity changes.
Figure 3. Measured fiberboard moisture content (%WME) vs actual moisture content (wt% water) at room temperature. The curve fit is valid between 6 and 40 %WME.

Figure 4. Relative humidity of air vs measured moisture content of fiberboard at room temperature.
Figure 5. Weight change of a 9975 package upper fiberboard subassembly during and following exposure to a 100% humidity environment.

Figure 6. Weight change of three 9975 package upper fiberboard assemblies with varying degree of isolation from the room environment.