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**SIXTH STATUS REPORT: TESTING OF AGED SOFTWOOD FIBERBOARD
MATERIAL FOR THE 9975 SHIPPING PACKAGE**

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Sixth Status Report: Testing of Aged Softwood Fiberboard Material for the 9975 Shipping Package

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Summary

Samples have been prepared from several 9975 lower fiberboard subassemblies fabricated from softwood fiberboard. Physical, mechanical and thermal properties have been measured following varying periods of conditioning in each of several environments. These tests have been conducted in the same manner as previous testing on cane fiberboard samples.

Overall, similar aging trends are observed for softwood and cane fiberboard samples, with a few differences. Some softwood fiberboard properties tend to degrade faster in some environments, while some cane fiberboard properties degrade faster in the two most aggressive environments. As a result, it is premature to assume both materials will age at the same rates, and the preliminary aging models developed for cane fiberboard might not apply to softwood fiberboard. However, it is expected that both cane and softwood fiberboard assemblies will perform satisfactorily in conforming packages stored in a typical KAC storage environment for up to 15 years.

Samples from an additional 3 softwood fiberboard assemblies have begun aging during the past year to provide information on the variability of softwood fiberboard behavior. Aging and testing of softwood fiberboard will continue and additional data will be collected to support development of an aging model specific to softwood fiberboard.

Background

Cane fiberboard wall sheathing is specified for thermal insulation and impact resistance in 9975 shipping packages. Softwood fiberboard manufactured by Knight-Celotex was approved as an acceptable substitute for transportation in 2008. Data in the literature [1] show a consistent trend in thermal properties of fiberboard as a function of temperature, density and/or moisture content regardless of material source. Thermal and mechanical properties were measured for un-aged softwood fiberboard samples, and found to be sufficiently similar to those of un-aged cane fiberboard to support the acceptance of 9975 packages with softwood fiberboard overpack into the K-Area Complex (KAC) Material Storage Area (MSA) for storage. The continued acceptability of aged softwood fiberboard to meet KAC storage requirements was the subject of subsequent activities.

This is an interim status report for experiments carried out per Task Technical and QA Plan [2], which is part of the comprehensive 9975 package surveillance program [3]. The primary goal of this task is to validate the preliminary assessment that Knight-Celotex softwood fiberboard is an acceptable substitute for cane fiberboard in the 9975 shipping package overpack, and to verify whether the long-term performance of these two materials in a storage environment is comparable.

Experimental Method

A lower fiberboard subassembly fabricated from softwood fiberboard for use in a 9975 shipping package was obtained from KAC. Samples were removed from this subassembly for conditioning and testing to track the potential degradation in physical, thermal and mechanical properties. Samples began aging in the following 4 environments in November 2008.

- 250F oven
- 215F oven
- 185F oven
- 185F, 30%RH

A second set of samples from the original softwood fiberboard assembly began aging in 2 additional environments in 2011: 160F 50%RH and 125F 70%RH. In 2014, additional samples from the original softwood assembly began aging in 2 more environments – 125F oven and 185F 70%RH. With this addition, aging of softwood samples was being conducted in all the environments used for cane fiberboard samples. Also in 2014, samples from 3 additional softwood fiberboard assemblies began aging in several environments to look for package-to-package variation. These additional assemblies came from 2 training packages (designated T4 and T5) and package 9975-06100 which was removed from service for destructive examination. The current test matrix now includes the following representation:

Softwood Source Assemblies for the Following Sample Types				
Environment	Physical Property	Compression	Thermal Conductivity	Specific Heat Capacity
250F, dry	Orig., T4, T5, 6100	Orig., T4, T5, 6100	Orig., T4, T5, 6100	Orig.
215F, dry	Orig., T4, T5, 6100	Orig.	Orig.	--
185F, dry	Orig., 6100	Orig., 6100	6100	--
125F, dry	Orig., 6100	Orig.	Orig.	--
185F, 70%RH	Orig., 6100	Orig., 6100	Orig., 6100	--
185F, 30%RH	Orig., T4, T5, 6100	Orig.	Orig., 6100	Orig.
160F, 50%RH	Orig., T4, T5, 6100	Orig., T4, T5, 6100	Orig., T4, T5, 6100	--
125F, 70%RH	Orig., 6100	Orig.	Orig.	--

The sample configurations and test methodologies are the same as used for aging and testing cane fiberboard samples [4, 5]. Samples for physical property measurements are approximately 2 inch cubes, and receive periodic measurement of weight and dimensions. Samples for compression testing are also approximately 2 inch cubes. A few of these samples are removed periodically for testing. Since the compression test is destructive, these samples are not returned to the conditioning environment.

Testing for thermal properties includes both thermal conductivity (per ASTM C518) and specific heat capacity (per ASTM C351). Thermal conductivity samples are approximately 7 x 7 x 1.3 inches. These samples are usually prepared in pairs: in each sample pair, one is oriented for axial heat flow, and the other is oriented for radial heat flow (relative to the package geometry). Thermal conductivity is measured at 2 mean temperatures – 25 and 50C (77 and 122F) – for all samples. Limited data has been collected at a mean temperature of 85C (185F) for samples in 185F and hotter aging environments.

Specific heat capacity samples are cylindrical, approximately 1 inch diameter and 1.5 inches high. Three of these samples are conditioned in each of 2 environments (250F oven and 185F 30%RH chamber) and tested periodically. Specific heat capacity is measured for each of two mean

temperatures: 25 and 52C (77 and 125F). Of these two temperatures, 125F provides the more reliable results, with less scatter among multiple trials.

Thermal and physical property samples were characterized before conditioning, and non-aged compression samples were tested without conditioning to document baseline behavior.

Results

The physical property samples were initially measured weekly, and are currently measured on an approximately biweekly basis. Typical data from the original softwood fiberboard assembly are shown in Figure 1 on a normalized basis (each datum is divided by its corresponding value after the first conditioning period) for each environment. This normalization allows for a direct comparison of degradation between samples with different starting values. The rates of change in the weight, density and dimensions of these samples are summarized in Tables 1 and 2 for all samples which have aged for ~300 days or more, and for samples which have aged for ~1100 days or more. Physical property samples from 9975-06100 have aged for less than 300 days, and do not yet show reliable degradation rates. Rates of change for cane fiberboard samples over similar aging periods are also shown in Table 1, for comparison.

Compression testing is performed with the load applied either parallel or perpendicular to the fiberboard layers. Typical stress-strain curves for softwood fiberboard samples tested in the parallel orientation are shown in Figure 2. Typical stress-strain curves for softwood fiberboard samples tested in the perpendicular orientation are shown in Figure 3.

Because of variation in the shape of the stress-strain curve from one sample to another, two metrics have been used to provide a comparison of compression test performance. For samples of both orientations, the area under the stress-strain curve up to a strain of 40% provides a metric that is roughly proportional to the energy absorbed by the material. In addition, samples tested in the parallel orientation experience an initial stress peak as the fiberboard layers start to buckle. This buckling strength provides a second metric for comparison of the parallel orientation samples. The energy absorption metric is summarized in Figure 4, along with comparable data for cane fiberboard samples. (The cutoff at 40% strain is an arbitrary value that captures most of the deformation likely to occur in an accident scenario while providing a consistent point of comparison across samples with potentially wide variation in behavior.)

Thermal conductivity data for each sample are presented in Figure 5 at a mean temperature of 25C. Similar trends are generally seen for each of the three test temperatures – 25, 50 and 85C – with some deviation seen at 85C for the most humid environments due to moisture migration during testing. A more complete comparison of thermal conductivity degradation rates for softwood and cane fiberboard samples is shown in Table 3. Degradation rates are not listed in Table 3 for samples with less than 32 weeks aging time. Since the baseline thermal conductivity varies for each sample, normalized data are shown in Figure 6 for two environments, and show the relative change from the first conditioned data point. Comparable normalized data for typical cane fiberboard samples are also shown in Figure 6.

Specific heat capacity results are summarized in Figure 7. Due to the degree of scatter in individual results, data from each trial for all 3 samples in a given environment are averaged for each conditioning period. Comparable data for cane fiberboard samples are also shown in Figure 7.

Discussion

No significant degradation has been observed in fiberboard assemblies from conforming packages (i.e. packages without excessive moisture and/or mold) examined following up to 7 years storage in KAC. The typical package stored in KAC contains a modest amount of moisture within the fiberboard assembly, and has an internal heat load significantly less than the 19 watt rating of the package. Most of the packages contain a cane fiberboard overpack assembly, although an increasing number contain softwood fiberboard (since cane fiberboard assemblies are no longer being produced).

The ambient temperature within the KAC MSA can vary seasonally, or due to changes in HVAC status. However, for a typical summertime ambient temperature of ~85F and an internal heat load of 10 watts or less, the maximum fiberboard temperature is expected to be about 115F for cane fiberboard. (This estimate is based on the 59F increase from ambient to the maximum shield temperature calculated for a 19 watt heat load in Reference 6.) With softwood fiberboard, the maximum fiberboard temperature would be about 2-3F higher, based on Reference 7. Within these packages, the warmer regions will tend to have lower moisture content, and the cooler regions will typically have slightly elevated moisture.

To date, packages removed from storage for destructive examination include 7 with cane fiberboard and 1 with softwood fiberboard. They had been held in storage for periods ranging from ~5 months to 7 years. The consistent trend indicates the storage environment is sufficiently mild to preclude significant degradation over this time period, although baseline data from these specific assemblies are not available for comparison. Properties measured on the one softwood fiberboard assembly are consistent with those measured on the cane fiberboard assemblies. In contrast, the environments used for accelerated aging of the test samples described in this report are more severe than typical KAC storage conditions. This difference is necessary in order to observe degradation and develop models for predicting service life in advance of unacceptable degradation occurring in KAC.

In analyzing the compression data, the energy absorption data for a given environment are extrapolated to the time for this metric to drop to 11 psi. This value has been shown to be the minimum to provide the needed protection for accident scenarios during storage [8]. This extrapolated time is summarized for both cane and softwood fiberboard samples in Table 4. Different estimates are developed for the two test orientations. The actual behavior of fiberboard within the 9975 package during a side impact event (loading primarily in the parallel orientation, but with some degree of constraint on the material) likely falls between projections for the two orientations. The utility of the data is limited for some environments due to limited duration of data (no more than 16 weeks aging) and the data scatter (giving an overall positive slope, indicating no degradation).

Overall, similar aging trends are observed to date for softwood and cane fiberboard samples, with most properties in most of the aging environments degrading at essentially the same rate for both softwood and cane fiberboard. Previous status reports [9] have identified that when differences occur between the two materials, the softwood fiberboard properties degrade faster than those of cane fiberboard in the elevated humidity environments, and the cane fiberboard properties degrade faster in the higher temperature dry environments. With the additional material that began aging in the past year, the current data set shows some exceptions to this trend (see Table 5). The instances of faster degradation of cane fiberboard occur in the two most aggressive environments (250F dry and 185F 70%RH), while instances of faster degradation of softwood fiberboard occur in the less aggressive environments. Since some of these cases involve less than 1 year aging time, further testing is needed to determine long-term trends. At this stage, it is sufficient to recognize that there are enough differences between the two materials to warrant separate degradation models for service life prediction.

Despite the modest differences between cane and softwood fiberboard, the present data and engineering judgment suggest that both cane and softwood fiberboard assemblies in conforming packages should perform satisfactorily for up to 15 years under typical KAC storage conditions.

Conclusions

Overall, similar aging trends are observed for softwood and cane fiberboard samples, with a few differences. There are modest differences between the two materials in several properties following aging in some of the environments. As a result, it is premature to assume both materials will age at the same rates, and the preliminary aging models developed for cane fiberboard might not apply to softwood fiberboard. However, it is expected that both cane and softwood fiberboard assemblies will perform satisfactorily in conforming packages stored in a typical KAC environment for up to 15 years.

Within the past year, additional softwood samples from additional source packages have been added to the test matrix, along with additional samples from the original softwood fiberboard assembly. This expanded the total test matrix to include all 8 environments for which cane fiberboard data exist. Aging and testing of softwood fiberboard samples in all environments will continue in order to support development of an aging model specific to softwood fiberboard.

References

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- [2] WSRC-TR-2008-00024, "Task Technical and Quality Assurance Plan for Testing to Support Acceptance into KAMS of Model 9975 Packages with Softwood Fiberboard Overpack (U)", January 2008. This reference was superseded in 2014 by SRNL-TR-2014-00057, "Task Technical and Quality Assurance Plan for Characterization and Surveillance of Model 9975 Shipping Package O-Rings and Fiberboard Materials", April 2014.

- [3] Historic reference: SRS Surveillance Program for Storage of Pu Materials in KAMS, WSRC-TR-2001-00286, Rev 3, December, 2006. Superseded by SRS Surveillance Program for Storage of Pu Materials in KAMS, WSRC-TR-2001-00286, Rev. 6, October 2011.
- [4] PVP2007-26114, "Properties of Fiberboard Overpack Material in the 9975 Shipping Package following Thermal Aging", W. L. Daugherty, Proceedings of PVP 2007 Conference, July 2007, ASME.
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- [7] M-CLC-K-00729, "Thermal Analysis of the 9975 Package with Softwood-Based Fiberboard during KAMS Facility Fire", N. K. Gupta, June 11, 2008.
- [8] SRNL-STI-2013-00020, "Status Report – Cane Fiberboard Properties and Degradation Rates for Storage of the 9975 Shipping Package in KAMS", W. L. Daugherty, January 2013.
- [9] SRNL-TR-2014-00069, "Fifth Status Report: Testing of Aged Softwood Fiberboard Material for the 9975 Shipping Package", W. L. Daugherty, April 2014.

Table 1. Weight and density decreases in softwood vs cane fiberboard over the stated aging period. Rates of change for softwood fiberboard are in bold if they are more than 0.3 %/yr outside the range observed for cane fiberboard samples.

Environment	Rate of Loss (%/yr) for		
	Original Softwood Assembly	Training Pkg Softwood Assemblies	Cane Fiberboard (multiple pkgs)
Weight change – ~300 days duration			
250F, dry	17.05, 17.31	15.57, 15.62	10.46 – 28.24
215F, dry	3.78, 3.94	3.65, 3.71	3.20 – 4.66
185F, dry	1.02, 1.02	--	1.38 – 2.88
125F, dry	-0.11 (gain)	--	-0.62 – -1.06 (gain)
185F, 70%RH	26.34	--	32.73 – 52.23
185F, 30%RH	7.27, 7.46	5.93, 6.14	3.49 – 4.43
160F, 50%RH	4.33, 4.47	3.72, 3.75	3.74 – 6.27
125F, 70%RH	1.27, 1.34	--	0.13 – 0.57
Weight change – ~1100 days duration			
250F, dry	11.42, 11.68	--	12.41 – 14.52
215F, dry	3.32, 3.48	--	3.01 – 3.76
185F, dry	1.24, 1.24	--	0.87 – 1.32
185F, 30%RH	5.66, 5.78	--	3.48 – 4.62
160F, 50%RH	3.25, 3.36	--	2.51 – 3.72
125F, 70%RH	0.90, 0.91	--	0.27 – 0.71
Density change – ~300 days duration			
250F, dry	6.41, 7.24	5.66, 6.37	5.60 – 12.76
215F, dry	-1.02 (gain), -0.52	-0.22 (gain), 0.40	-1.02 (gain) – 2.43
185F, dry	-2.53 (gain), -1.92	--	0.24 – 2.08
125F, dry	-0.30 (gain)	--	-0.89 – -0.44 (gain)
185F, 70%RH	17.35	--	13.78 – 27.56
185F, 30%RH	1.51, 1.72	1.50, 2.23	2.03 – 3.69
160F, 50%RH	2.77, 2.77	-0.15 (gain), 0.50	1.62 – 2.79
125F, 70%RH	1.02, 1.49	--	0.21 – 0.95
Density change – ~1100 days duration			
250F, dry	5.53, 6.49	--	7.05 – 8.33
215F, dry	0.87, 0.96	--	-0.06 (gain) – 1.90
185F, dry	0.02, 0.11	--	-0.53 (gain) – 0.72
185F, 30%RH	3.01, 3.05	--	1.17 – 2.26
160F, 50%RH	1.53, 1.97	--	1.10 – 1.70
125F, 70%RH	0.21, 0.24	--	-0.13 (gain) - 0.17

Table 2. Dimensional decreases in softwood vs cane fiberboard over the stated aging period. Rates of change for softwood fiberboard are in bold if they are more than 0.3 %/yr outside the range observed for cane fiberboard samples.

Environment	Rate of Loss (%/yr) for		
	Original Softwood Assembly	Training Pkg Softwood Assemblies	Cane Fiberboard (multiple pkgs)
Height change – ~300 days duration			
250F, dry	8.20, 8.67	7.07, 7.82	2.75, 7.78
215F, dry	3.01, 5.31	1.98, 2.26	0.84, 3.23
185F, dry	1.48, 2.47	--	-0.12 (gain) – 1.21
125F, dry	0.08	--	-0.37, -0.13 (gain)
185F, 70%RH	6.13	--	10.73 – 20.51
185F, 30%RH	3.69, 3.91	2.49, 3.35	1.28 – 1.64
160F, 50%RH	1.34, 1.62	2.30, 3.46	1.62 – 2.79
125F, 70%RH	0.09, 0.12	--	-0.23 (gain) – 0.26
Height change – ~1100 days duration			
250F, dry	4.56, 5.09	--	3.59 – 6.28
215F, dry	1.53, 1.64	--	1.06 – 1.54
185F, dry	0.61, 0.77	--	0.35 – 0.63
185F, 30%RH	1.75, 1.83	--	1.28 – 1.76
160F, 50%RH	0.86, 1.25	--	0.82 – 1.35
125F, 70%RH	0.32, 0.32	--	-0.01 (gain) – 0.16
Length, Width change – ~300 days duration			
250F, dry	0.28 , 2.44	0.82 – 1.89	0.92 – 4.11
215F, dry	0.52, 1.13	0.63 – 0.95	0.001 – 1.69
185F, dry	0.26, 1.23	--	0.03 – 0.78
125F, dry	-0.01 (gain), 0.12	--	-0.33(gain) – 0.31
185F, 70%RH	2.00, 2.69	--	6.28 – 8.38
185F, 30%RH	0.78, 1.26	0.57 – 0.92	-0.03 (gain) – 0.45
160F, 50%RH	-0.003 (gain) , 0.18	0.16 – 0.65	0.46 – 1.03
125F, 70%RH	-0.47 (gain), 0.18	--	-0.43 (gain) – 0.37
Length, Width change – ~1100 days duration			
250F, dry	0.90 – 1.97	--	1.04 – 3.34
215F, dry	0.43 – 0.56	--	0.28 – 1.38
185F, dry	0.23 – 0.28	--	0.02 – 0.52
185F, 30%RH	0.55 – 0.81	--	0.35 – 0.59
160F, 50%RH	0.25 – 0.35	--	0.26 – 0.69
125F, 70%RH	0.13 – 0.26	--	0.07 – 0.17

Table 3. Thermal conductivity (at 25C) changes in softwood vs cane fiberboard over the stated aging period. Rates of change for softwood fiberboard are in bold if they are more than 0.3 %/yr outside the range observed for cane fiberboard samples.

Orientation	Environment	Aging Period (weeks)	Softwood Fiberboard	Cane Fiberboard
			Rate of Decrease (%/yr)	Rate of Decrease (%/yr)
Axial	250F, dry	185	5.26 *	7.08 to 7.56
	215F, dry	302	1.76	1.80 to 2.05
	125F, dry	32	2.39	--
	185F, 70%RH	48	14.48	--
	185F, 30%RH	250	2.46	2.05 to 2.44
	160F, 50%RH	197	2.27	1.40 to 2.06
	125F, 70%RH	200	0.34	-0.46 (gain) to 0.01
Radial	250F, dry	201	8.28 *	9.32 to 10.77
		48	13.26, 14.35	14.86 to 16.46
	215F, dry	302	2.16	2.13 to 2.30
	125F, dry	32	-0.92 (gain)	-0.64 to -0.25 (gain)
	185F, 70%RH	48	25.23 *	--
	185F, 30%RH	250	2.89	1.97 to 2.98
	160F, 50%RH	197	1.71	1.49 to 2.81
		48	1.41, 1.58	2.39 to 3.59
	125F, 70%RH	200	-0.76 (gain)	-1.07 (gain)

* These samples have been retired, having reached a state of significant degradation (typically delamination and extreme fragility).

Table 4. Extrapolated estimates of the time for energy absorption (represented by the area under the stress-strain curve up to 40% strain) to degrade to 11 psi. Estimates are based on an exponential fit to the data for each source package. Values are provided for parallel orientation / perpendicular orientation.

	125F, 70%	160F, 50%	185F, 30%	185F, 70%	125F, dry	185F, dry	215F, dry	250F, dry
Softwood fiberboard- time to degrade to 11 psi, (yrs)								
Original	6.42 / **	1.36 / 8.83	1.16 / 7.15	0.29 / 0.56	* / *	14.54 / 30.56	* / *	1.97 / 2.82
Cane fiberboard – time to degrade to 11 psi, yrs								
LD1	26.1 / *	2.6 / 14.4	1.9 / 8.9	0.34 / 0.38	241 / **	10.9 / 105	4.5 / 12.1	1.6 / 2.6
LD2	5.4 / *	5.9 / *	--	0.49 / 0.37	** / **	19.7 / 1459	7.7 / 8.4	1.1 / 1.5
MSC	* / --	* / *	2.0 / 2.0	0.34 / 0.42	** / **	1102 / **	2.7 / 16.2	1.6 / 2.1
New	** / **	5.4 / 24.2	3.3 / 11.4	0.44 / 0.58	--	** / **	--	1.0 / 3.2

* Data is not available for these source packages / environments beyond 16 weeks exposure.

Extrapolation from this short range is not considered reliable.

** Data for these source packages / environments has a positive slope, which extrapolates to an infinite time to reach 11 psi.

Table 5. Comparison of softwood and cane fiberboard behavior. The listing of one material indicates that material degraded at least 0.3 %/yr faster for the particular environment and property.

	125F 70% RH	160F 50% RH	185F 30% RH	185F 70% RH	125F Dry	185F Dry	215F Dry	250F Dry
Physical Properties								
Weight	~ Same	~ Same	SW (2)	Cane	SW (1)	~ Same	~ Same	Cane
Height	~ Same	~ Same	~ Same	Cane	~ Same	~ Same	~ Same	~ Same
Length. Width	~ Same	~ Same	~ Same	Cane	~ Same	~ Same	~ Same	~ Same
Density	~ Same	~ Same	SW (2)	~ Same	~ Same	~ Same	~ Same	Cane
Compression Strength								
Area under Stress-Strain Curve, parallel	~ Same	SW (1)	SW (2)	~ Same	NA	~ Same	NA	~ Same
Area under Stress-Strain Curve, perpendicular	~ Same	SW (1)	~ Same	~ Same	NA	SW (1)	NA	~ Same
Thermal Conductivity								
Axial	SW (1)	~ Same	~ Same	~ Same	~ Same	NA	~ Same	Cane
Radial	SW (1)	~ Same	~ Same	~ Same	~ Same	NA	~ Same	Cane
Specific Heat Capacity								
	NA	NA	~ Same	NA	NA	NA	NA	~ Same

Softwood (1) - Softwood samples degrading faster than cane samples

Softwood (2) - Softwood samples degrading faster than cane samples (but weakest cane package not aged/tested in this environment)

Cane - Cane samples degrading faster than softwood samples

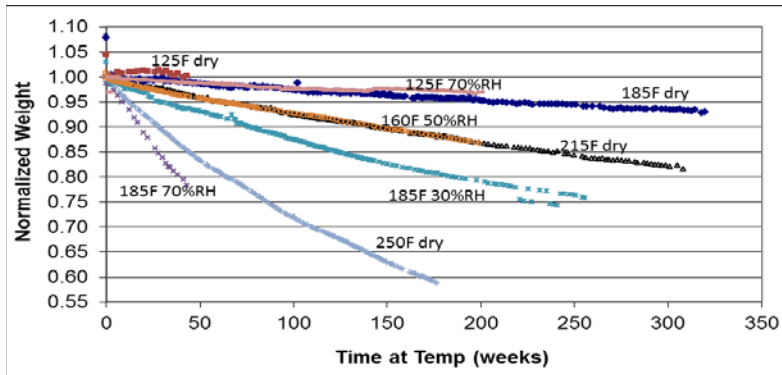
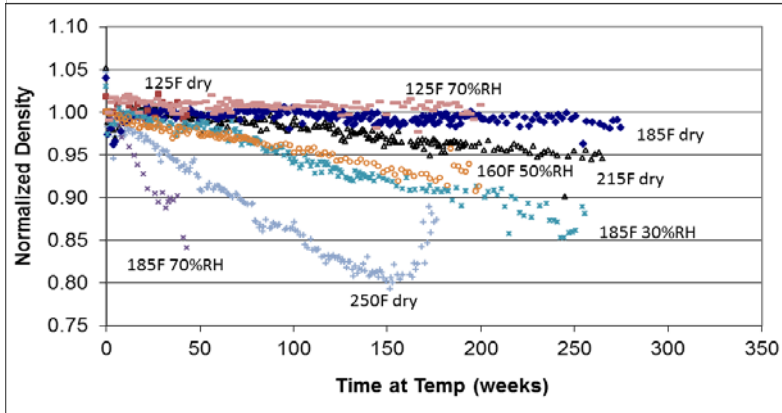
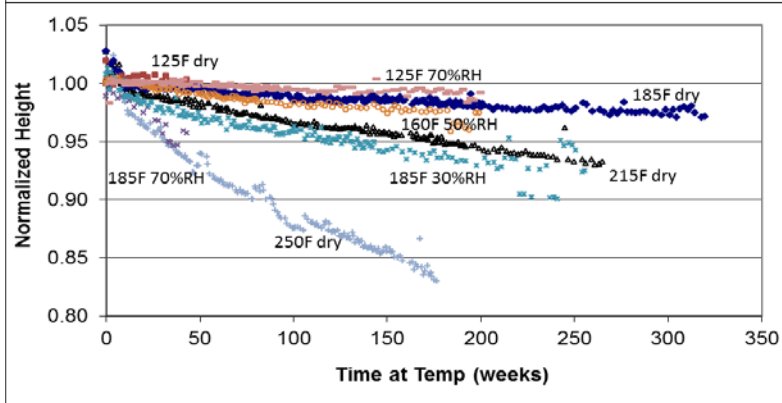


Figure 1. Normalized data for softwood fiberboard physical property samples.

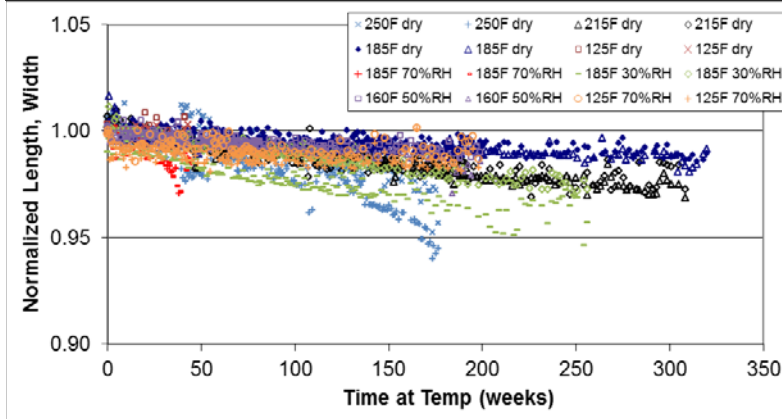
(a) Weight change



(b) Density change



(c) Height change



(d) Length / width change

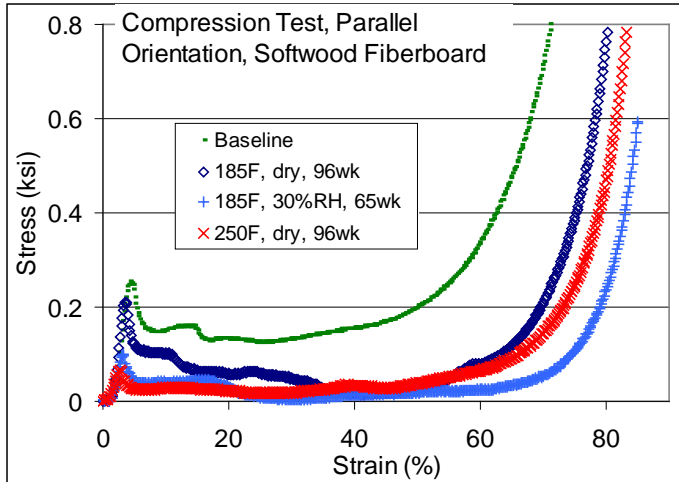


Figure 2. Typical compression stress-strain curves for softwood fiberboard samples, parallel orientation

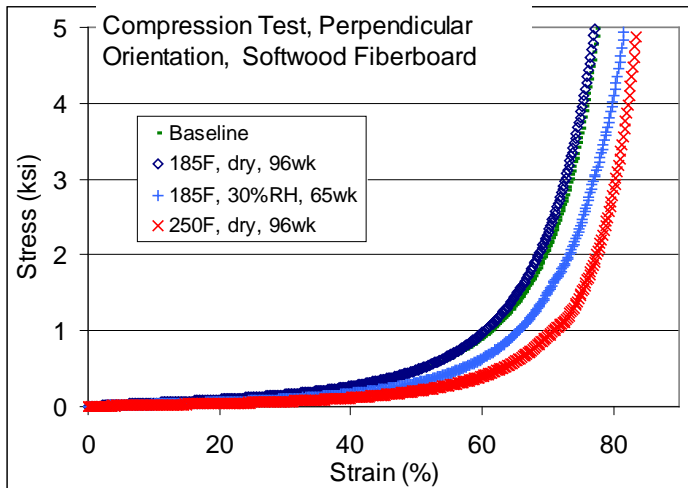


Figure 3. Typical compression stress-strain curves for softwood fiberboard samples, perpendicular orientation

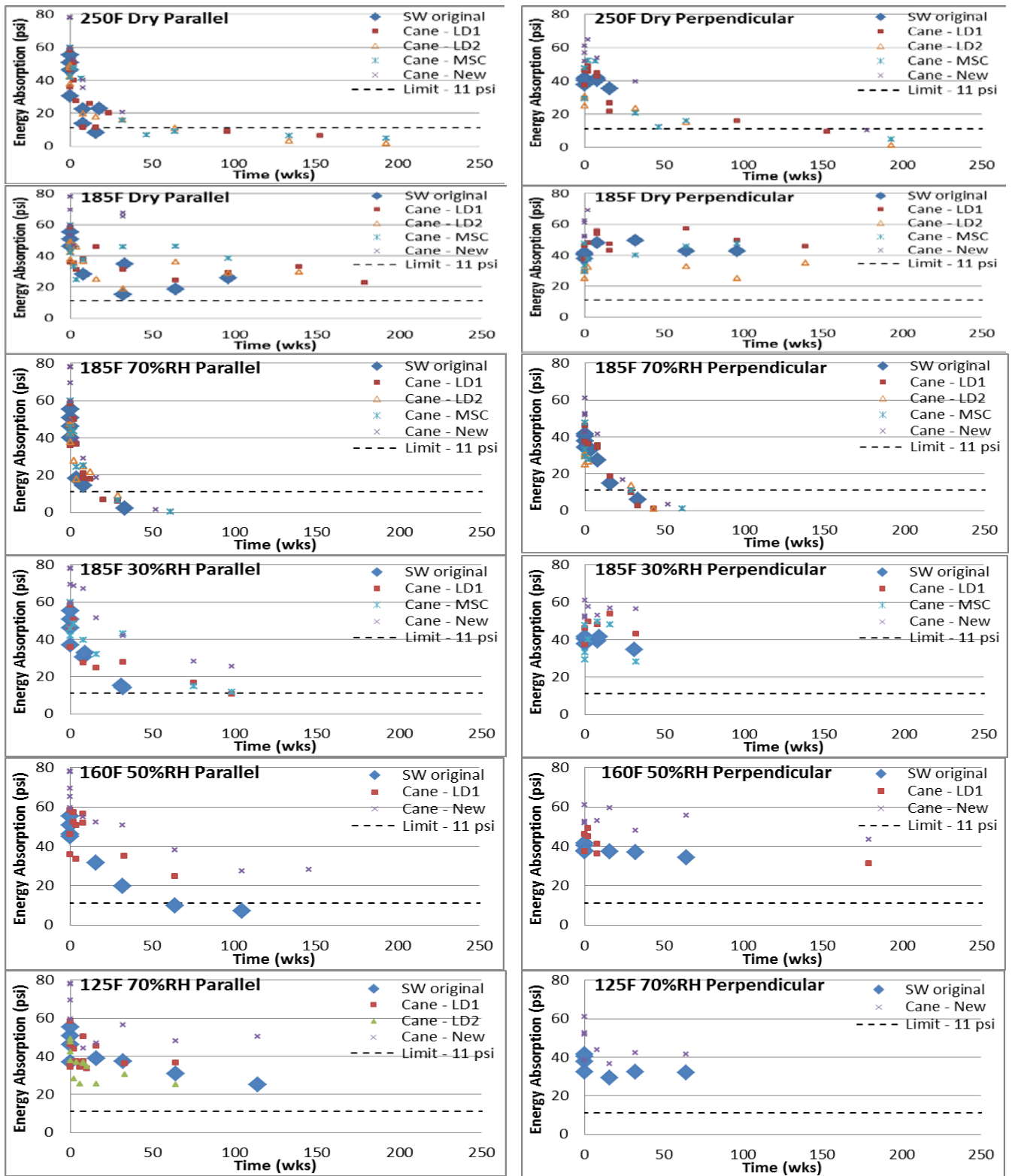


Figure 4. Energy absorption of compression test samples (represented by area under curve to 40% strain) for parallel (left column) and perpendicular (right column) orientation samples.

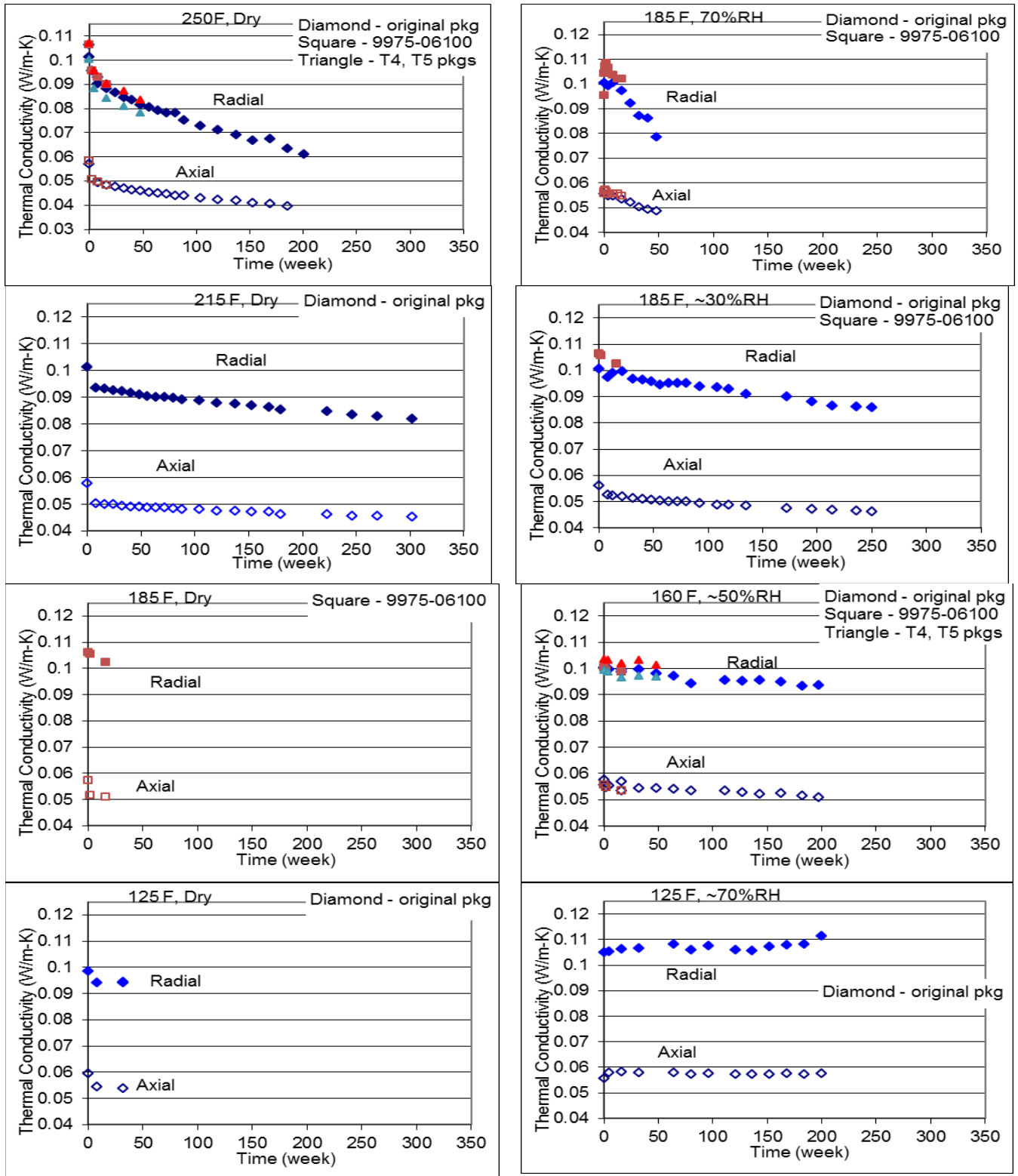
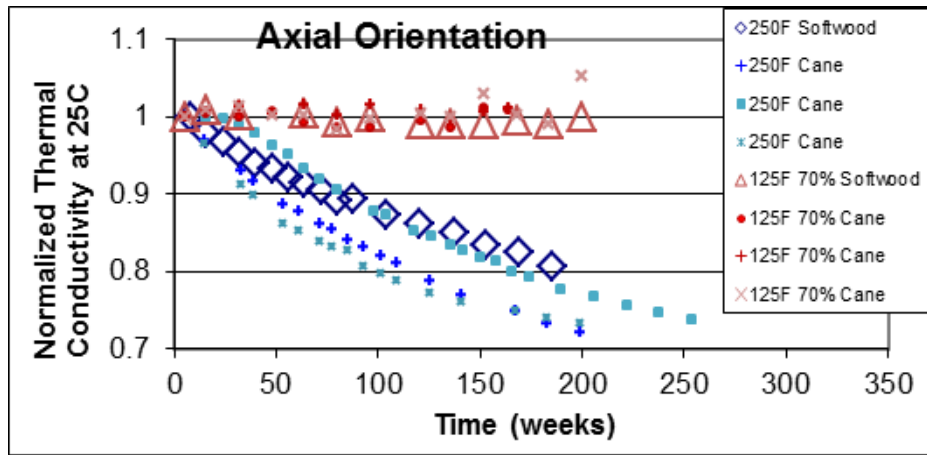
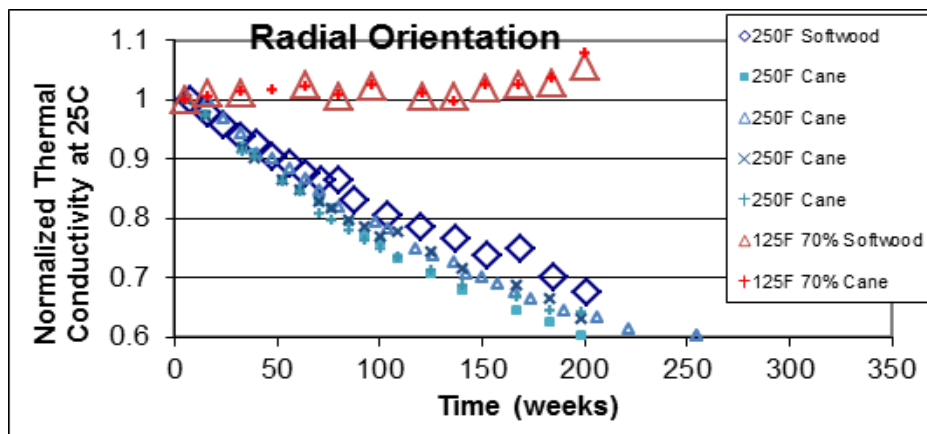


Figure 5. Thermal conductivity data for softwood fiberboard samples conditioned in the indicated environments



(a) axial orientation



(b) radial orientation

Figure 6. Normalized thermal conductivity data for softwood fiberboard compared to cane fiberboard in two environments (125F 70%RH and 250F dry). A more complete comparison of softwood and cane fiberboard thermal conductivity degradation rates is provided in Table 2.

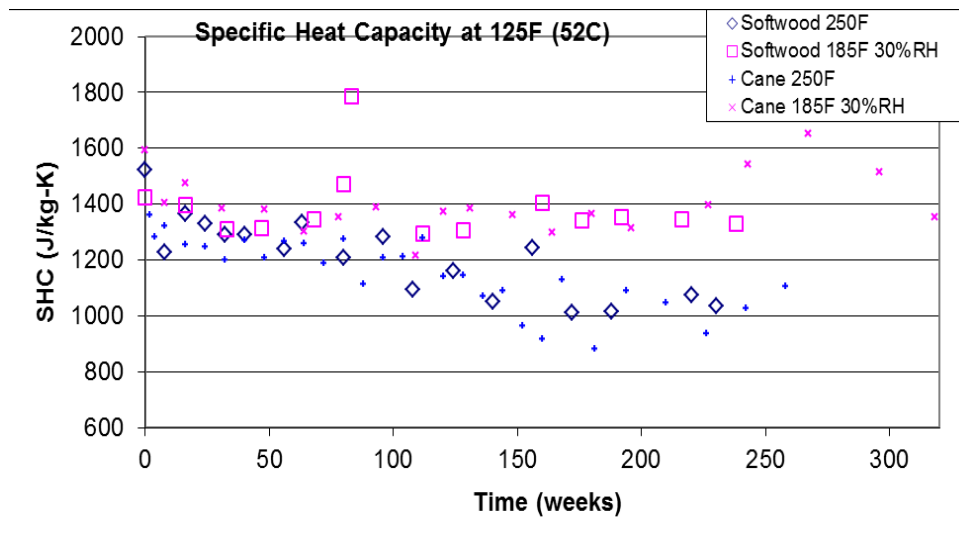


Figure 7. Specific heat capacity data for softwood fiberboard at a mean temperature of 52C, compared with cane fiberboard

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