INVESTIGATION OF SHEET MOLDING COMPOUND MADE WITH SOY-BASED AND PETROLEUM-BASED RESINS

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Abstract

Plaques fabricated from sheet molding compound (SMC) with soy-based resins in both glass fiber-reinforced and carbon fiber-reinforced versions are compared with the equivalent SMC with petroleum-based resins. Since soy-based resins are less sensitive to the price of petroleum than petroleum-based resins, these materials represent potential cost savings to the automotive industry if the price of petroleum continues to increase, as well as providing opportunities to decrease overall carbon dioxide emissions. Soy beans are also a renewable resource. Material thermal properties including dynamic mechanical analysis (DMA) and coefficient of linear thermal expansion (CLTE) are evaluated, as are mechanical properties including tensile and compressive characterizations. The effect of humidity aging was evaluated by moisture absorption, as well as residual tensile and compressive properties. For as-received properties, the glass-reinforced version of the soy-based material is found to be similar in performance to the petroleum-based material. However, the carbon-reinforced soy resin material has lower mechanical properties than the petroleum-based SMC, probably due to a lack of fiber-matrix adhesion. In humidity aging, the petroleum based materials absorbed less moisture than the soy-based, although the relative property loss caused by humidity aging was similar for the petroleum-based and the soy-based materials.

Introduction

There are many reasons to decrease the use of petroleum and petroleum-based products in all facets of our lives. These include environmental concerns, such as decreasing carbon dioxide emissions, as well as economic reasons as the price of petroleum continues to increase. A great deal of attention in the US automotive industry has been focused on decreasing petroleum use by lightweighting our vehicles, using advanced materials such as lower density or higher strength metals and composites. This addresses the overall consumption of gasoline, one of the primary petroleum products. A significant amount of this effort is focused through the Automotive Composites Consortium (ACC), which is a collaboration of General Motors, Ford, and DaimlerChrysler, doing pre-competitive research on structural and semi-structural composites, and working in conjunction with the US Department of Energy.

The ACC is also very interested in biocomposites, both natural fibers and soy-based resins. Not all of our petroleum use is for gasoline. Most of the polymeric resins used in the composite materials, which can lower the vehicle mass, are themselves based on petroleum. Switching a portion of these petroleum-based resins to resins based on soy oil is another means of decreasing our overall petroleum usage. While decreasing the need for imported petroleum, the use of soy-based resins also decreases the overall carbon dioxide emissions, since soybeans consume carbon dioxide when growing. Recent projections by the United Soy Board, an association of soybean growers, suggest that soy-based resins will be similar in price to petroleum-based resins (at 2006 prices) when stable production volumes of the soy-based materials are reached, and should be less expensive as petroleum continues to increase in price [1].

Soy-based resins are currently used by John Deere in body panels for harvesters, both in SMC and in Reaction Injection Molded parts (RIM). These are appearance panels for harvesters and tractors, although the appearance standards for these applications are much looser than automotive class A. The SMC resin is supplied by Ashland, and is cost neutral to the petroleum-based resin. The RIM resin is from Bayer. This has a small cost premium (less than 5%) which the molder does not pass on to Deere. While this application demonstrates the utility of soy-based resins, and is cost-neutral, it is also seen as good customer relations for John Deere, as it uses the soybeans that its customers grow.

The Ford Motor Company has done considerable work with soy-based foams for seating applications, partnering with the Lear Corporation and Bayer Corporation. These foams meet the materials requirements for head rests as well as seats with content of soy ranging from less than 5% to 20% of the total foam weight.

Other research into soy resins has included structural urethanes [2-4], urethane foams [5], epoxy systems [6], and polyester liquid molded resin [7], as well as polyester SMC materials [8]. In addition, "fully environmental-friendly" systems which include natural fibers and nano-clay reinforcements have been studied [9].

While composites made from soy-based resins may be a good means of providing ecologically responsible and cost effective materials, it is necessary to determine their mechanical and thermal properties, as well as resistance to automotive environments. The project reported herein describes the characterization of soy-based SMC materials, as part of the work of the ACC Materials Working Group. For this project, Ashland has supplied four sets of SMC panels, both glass-fiber and carbon-fiber reinforced, and both soy-based and petroleum-based resins. These panels have been evaluated for mechanical properties, as well as thermal properties and resistance to humidity. This report concerns the as-molded mechanical and thermal properties, as well as humidity aging, and the properties thereafter.

Experimental

Materials

The SMC panels were supplied by Ashland, and were compounded and molded in their laboratories. The formulations for these panels are shown in Table I. The formulations with the soy-based resins were said to be the same as with the petroleum-based resins, but were different for glass and carbon SMC.

Table I. Formulations for the SMC as supplied by Ashland.

Resin	Fiber	Wt%	Filler	pph
Petroleum-based unsaturated polyester (UPE)	Glass	30	W4 $CaCO_3$	150
Soy-based	Glass	30	W4 $CaCO_3$	150
Petroleum-based vinyl ester (VE)	Carbon	54	Nano-filler	1.5
Soy-based	Carbon	54	Nano-filler	1.5

Testing

Testing was according to the ACCM protocol [10], with the exception of sample placement. The panels supplied were 12 inches square, so the standard 24 inch square template could not be used. The template used for the 0º samples is shown in Figure 1. The 90º samples are cut by simply rotating the template 90º. Panels were tested both as received (AR) and after humidity aging (HA) for tensile and compressive properties. In addition, coefficient of linear thermal expansion (CLTE) and thermal mechanical behavior via dynamic mechanical analysis (DMA) were determined for the AR samples.

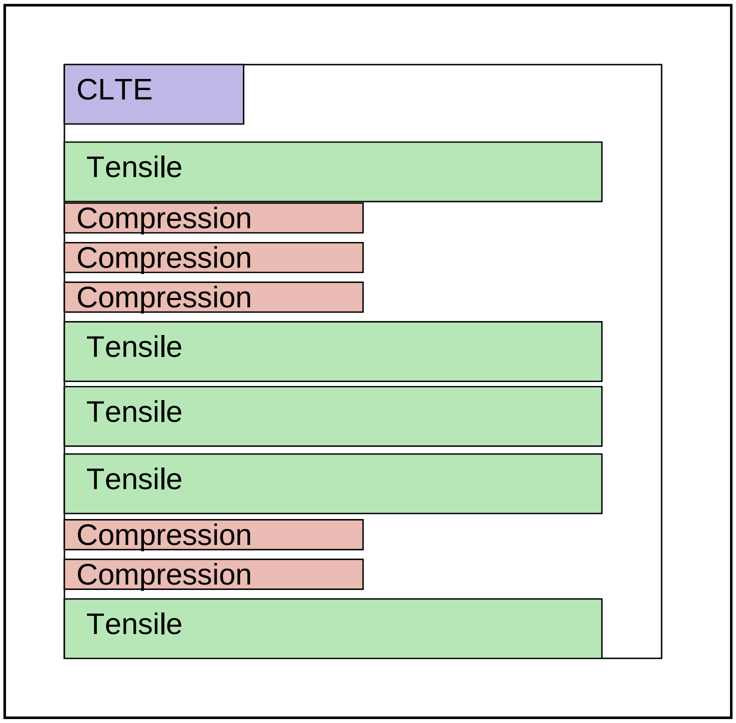

Figure 1. Template for samples in the 0º direction. Samples at 90º are positioned by rotating the template 90º.

At the time of testing the AR samples, we did not have an extensometer suitable for the compression testing, so only stress was measured for the compression samples. We acquired such an extensometer before testing the HA samples, so we are reporting the compressive modulus and strain for them, although there are no comparative AR data. Testing was done in both directions for AR samples. However, since we could not be certain of the orientation of the panels given to us, we elected to test only one direction for HA, and to compare this with the average of the two directions for the AR panels.

Samples cut for HA tensile and compression testing were first dried in a vacuum oven at 40ºC and 30 inches of mercury for 360 hours, at which time their weight loss had leveled off. After the vacuum oven drying, the samples were placed in a humidity chamber at 40ºC and 90%RH for 2500 hours. Weight measurements were taken daily throughout the drying and subsequent humidity aging.

Results and Discussion

Thermal Analysis

DMA testing was done for the four AR samples, with the results given in Table II. DMA is used to show the behavior of the modulus of a material with temperature. T_{75} is the temperature at which the storage modulus falls to 75% of its value at 25ºC. T_{onset} is one measure of the T_g, and is the temperature measured at the slope change of the storage modulus curve. T_{loss} is the T_g measured as the peak of the loss modulus curve.

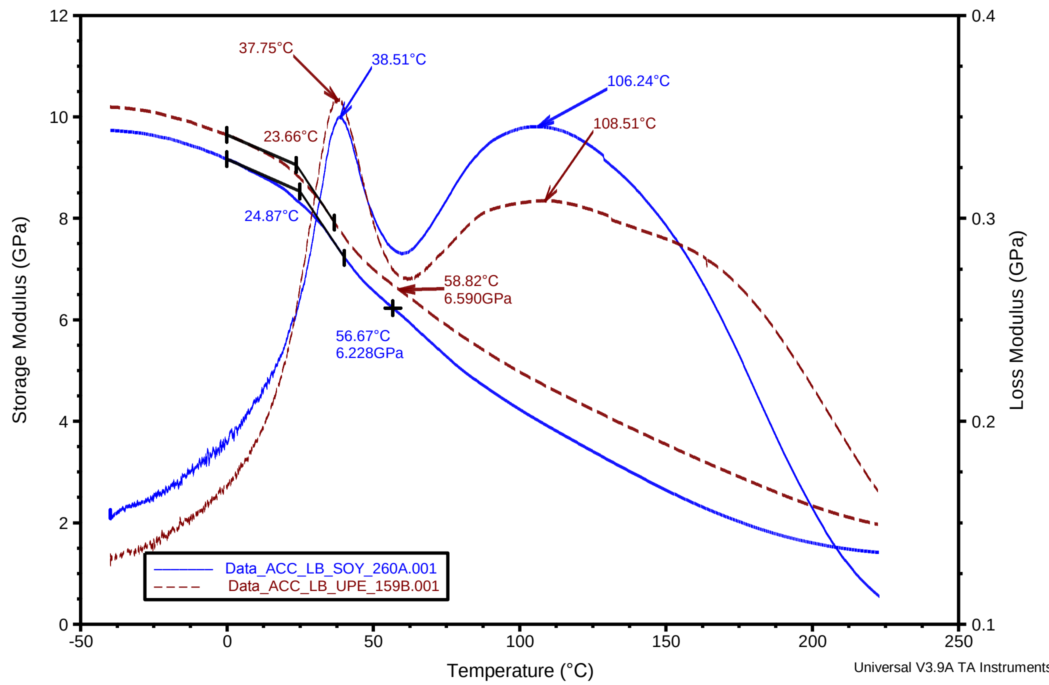

Figure 2. A DMA comparison of petroleum-based and soy-based glass-reinforced SMC.

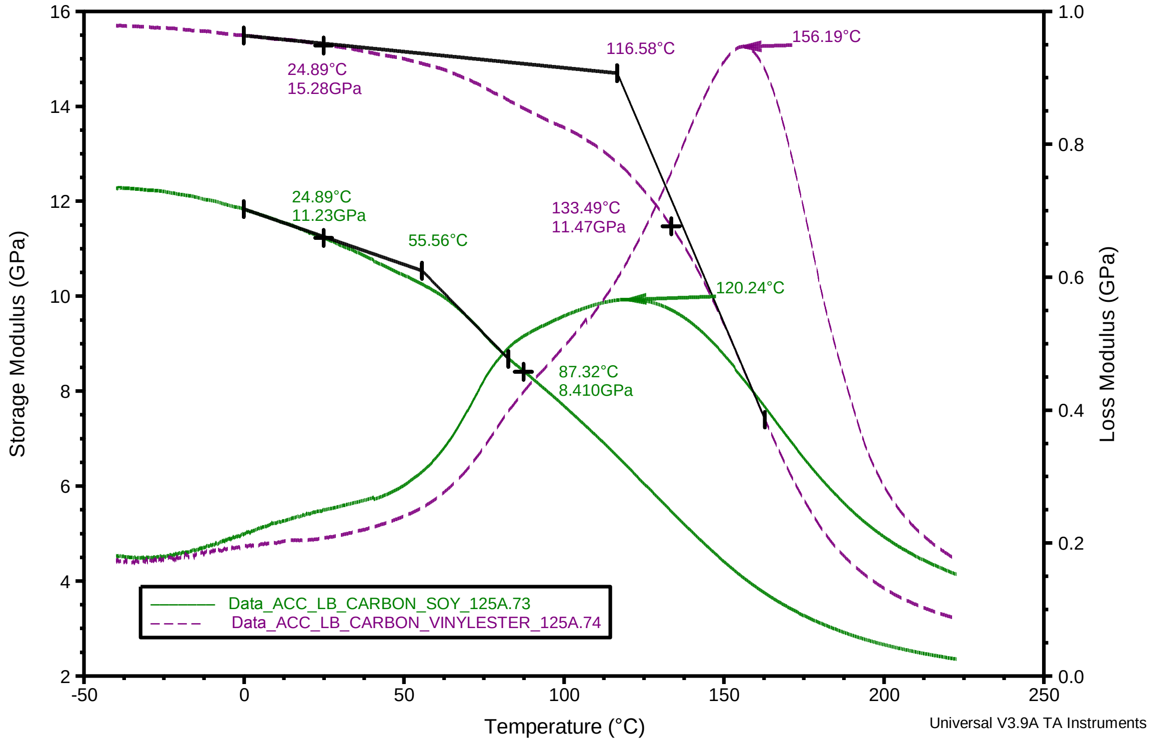

Figure 3. A DMA comparison of petroleum-based and soy-based carbon-reinforced SMC.

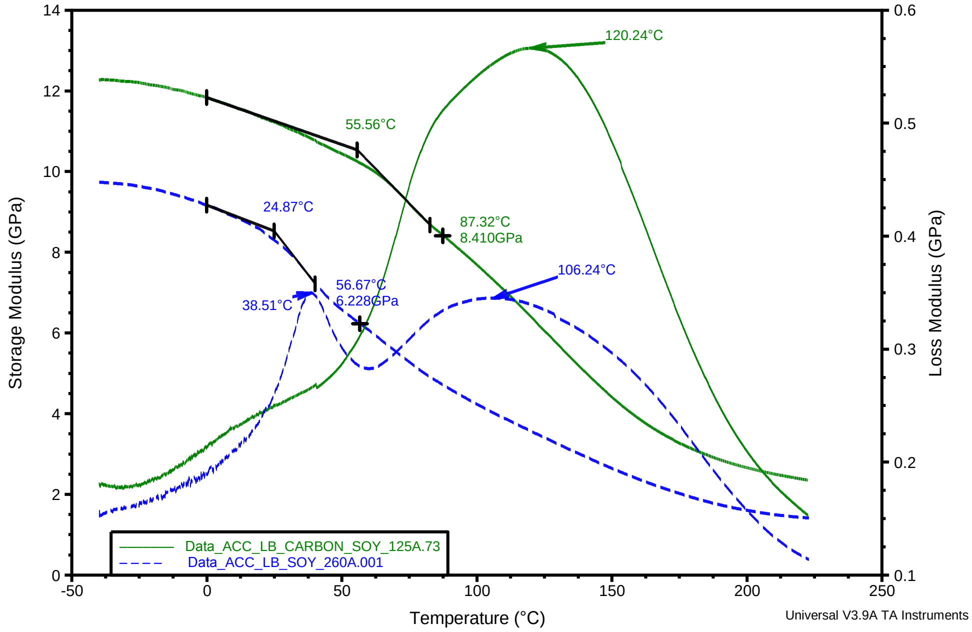

Figure 4. A DMA comparison of glass-reinforced and carbon-reinforced soy-based SMC.

Table II. Thermal transitions in the DMA curves for the four materials. Values are in °C.

	Glass/Soy	Glass/UPE	Carbon/Soy	Carbon/VE
$\mathbf{T_{75}}$	57	59	87	133
$\mathbf{T_{onset}}$	25	24	56	117
$\mathbf{T_{loss}}$	38, 106	38, 108	120	156

Figures 2 through 4 show the DMA curves. Figure 2 compares the soy-based and petroleum-based glass-reinforced SMC materials. The two materials have very similar thermal behavior, with the transitions being within 2° of each other. The loss modulus curve for both materials shows two distinct peaks. This is probably due to a low-profile agent or another lower molecular weight, phase-separated resin component, although the resin formulations are not available to us. This also convolutes the storage modulus curves, with the T_{onset} being determined by the first transition, and thus being quite low. The second loss modulus peak is a bit narrower in the soy-based formulation, which usually indicates denser crosslinking.

Figure 3 compares the two carbon-reinforced materials. In both formulations, there is only one peak in the loss modulus curve, indicating no phase-separated resin component. The vinyl ester resin is formulated for higher temperature properties, with a T_{loss} of 156°, and a much narrower peak than the soy-based material. Figure 4 compares the glass-reinforced and carbon-reinforced soy-based materials. While the width of the carbon loss modulus curve and the higher temperature portion of the glass curve are similar, the carbon-reinforced material peaks at a higher temperature. This may be because of differences in the resin formulations used for the two reinforcements, or may be because the carbon reinforcement mitigates the effect of the temperature.

CLTE results are shown in Table III and Figures 5 and 6. The CLTE is measured at three temperature ranges, -30º to 30ºC, 30º to 80ºC, and 80º to 120ºC, reflecting both what the material may see in normal usage and the possible conditions in manufacturing. Table III gives the CLTE values for these ranges and an overall value from -40º to 160ºC, as well as the correlation coefficient for the overall value. This correlation coefficient can be seen as a measure of the degree of change in the CLTE across the temperature range.

Table III. Values of CLTE for SMC materials. Materials were tested at both 0º and 90º orientations. Units are 10^6 x $^oC^{-1}$.

	Carbon/Vinyl Ester		**Carbon/Soy**		**Glass/UPE**		**Glass/Soy**	
	0º	**90º**	**0º**	**90º**	**0º**	**90º**	**0º**	**90º**
-30º to 30º	6.97	8.32	7.39	9.96	19.80	19.63	22.30	21.30
30º to 80º	3.9	5.3	4.08	2.77	8.76	7.75	5.46	5.93
80º to 120º	3.48	5.88	2.74	1.54	8.15	6.73	5.11	5.8
Overall	4.49	6.07	4.67	4.39	11.58	10.08	10.31	10.36
r^2	0.94	0.98	0.94	0.83	0.95	0.94	0.88	0.90

Figure 5 shows the linear expansion of two samples of the same material with temperature, normalized for sample length. The CLTE is the slope over a given temperature range. The material shown is the soy-based, carbon-reinforced SMC, cut at 0º and 90º orientations. Notice that the slope at the lower temperatures is significantly sharper than at the higher temperatures. This is the case in general for these materials, as Figure 6 illustrates. Figure 6 compares the CLTE values in the different temperature ranges. Another trend in Figure 6 and Table III is the greater expansion of the glass-reinforced materials, compared to the carbon-reinforced. Two factors are likely affecting this: the resin composition and the stiffness of the fibers. As shown in the DMA results above, the glass-reinforced SMC has a marked low-temperature transition not seen in the carbon-reinforced material, which will be a significant source of expansion. Any resin expansion, however, is constrained by the reinforcing fibers. The carbon fibers are much stiffer than the glass fibers, of course, and thus better able to constrain the resin expansion. The soy-based SMC's are quite similar to the petroleum-based materials in their overall CLTE values. In the lower temperature range, the soy-based resins are slightly higher, but in the two upper ranges they are slightly lower.

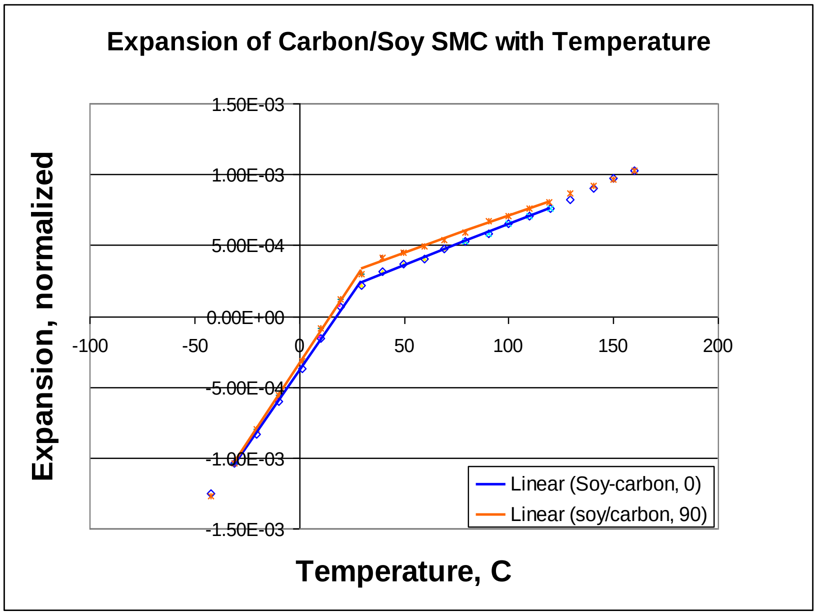

Figure 5. Expansion of carbon-reinforced, soy-based SMC, normalized for sample length, as a function of temperature in ºC. The CLTE is the slope of the line over a given temperature range. Data is shown for both 0º and 90º orientation.

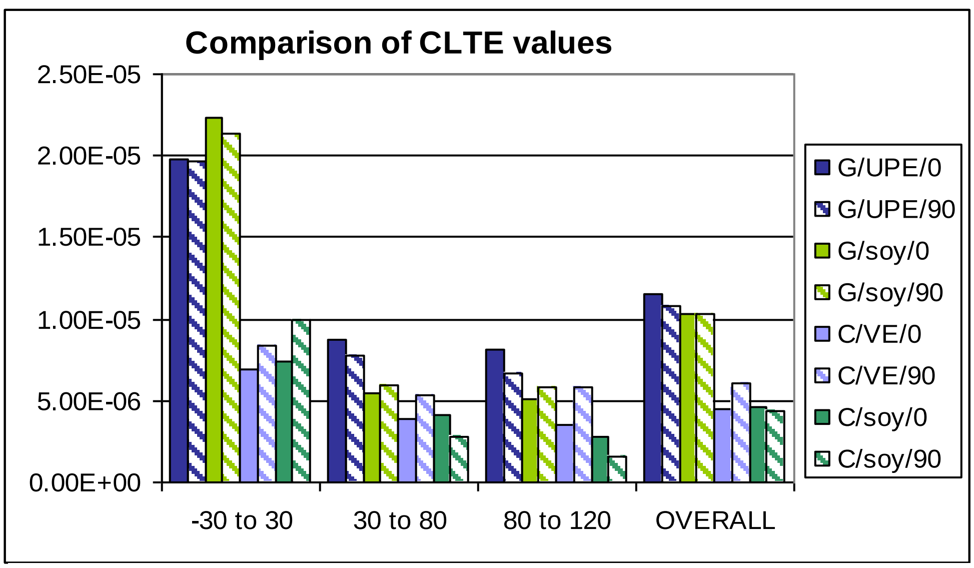

Figure 6. Comparison of CLTE values for petroleum-based and soy-based SMC materials, by temperature range in ºC. Samples are glass-reinforced or carbon-reinforced SMC, with either petroleum-based or soy-based resin. Values for 0º and 90º orientations are shown.

Mechanical Properties

The tensile properties for 0º and 90º orientation are given in Table IV, and the compression properties are in Table V. Figures 7, 8, 9, and 10 show the tensile stress, modulus, and strain and compressive stress values, respectively, for the four materials, at both 0º and 90º orientation.

Table IV. Tensile properties for petroleum-based and soy-based SMC. Values in parentheses are coefficients of variation (COV), the standard deviation divided by the mean, expressed as percent.

	stress (MPa)			modulus (GPa)			strain (%)		
	0º	**90º**	**avg**	**0º**	**90º**	**avg**	**0º**	**90º**	**avg**
glass/UPE	99 (7%)	97 (6%)	98	12.8 (8%)	11.3 (5%)	12.0	1.45 (6%)	1.40 (20%)	1.43
glass/soy	91 (10%)	87 (14%)	89	11.4 (5%)	10.6 (4%)	11.0	1.41 (16%)	1.47 (16%)	1.44
carbon/VE	223 (8%)	176 (13%)	200	41.3 (10%)	46 (14%)	43.6	0.39 (18%)	0.67 (22%)	0.53
carbon/soy	162 (6%)	125 (17%)	144	36.9 (14%)	30.5 (8%)	33.7	0.46 (17%)	0.48 (17%)	0.47

The ratio of 0º and 90º values given in Table VI shows the anisotropy, or degree to which the material is not random, of the composite. The glass materials show fairly low anisotropy, with little difference between the petroleum-based UPE material and the soy-based resin. The carbon materials show considerably more anisotropy, with the soy-based resin similar or slightly less anisotropic than the petroleum-based VE. This increased anisotropy may be because of filamentization of the carbon strands constraining the flow during molding. Carbon fibers are not currently engineered for SMC-type materials, where flow is important.

Table V. Compressive stress properties forpetroleum-based and soy-based SMC. Values in parentheses are COV's.

	Compressive Stress (MPa)		
	0º	**90º**	**avg**
glass/UPE	169 (8%)	174 (8%)	172
glass/soy	155 (7%)	152 (9%)	154
carbon/VE	303 (11%)	245 (4%)	274
carbon/soy	212 (12%)	185 (21%)	199

Table VII gives the ratio of the material properties for the petroleum-based resins to the soy-based resins. For the glass-based materials, the UPE performs, on average, 8% better than the soy-based resin. With COV's ranging from 4% to 20% (Tables IV and V) and averaging 9%, the 8% advantage for the petroleum-based material is not significant, and the indications are that the use of soy-based resins for glass-reinforced SMC is very promising.

For the carbon fiber SMC, however, the VE resin is 52% better than the soy-based resin. The COV's range from 4% to 22%, and average 14%. For the carbon materials, the soy-based resin does not perform equivalently to the petroleum-based. However, the adhesion of carbon fibers to non-epoxy resins is not well understood in the industry, and it is unlikely that the carbon fibers were optimized for the soy-resin. It will require considerable work with both the resin suppliers and the carbon fiber suppliers to understand and control the fiber-matrix adhesion.

Table VI. Ratio of 0º and 90º properties for soy and petroleum based composites, indicating degree of anisotropy.

		UPE/ Glass	Soy/ Glass	VE/ Carbon	Soy/ Carbon
Tensile	Modulus	1.13	1.08	1.11	1.21
	Stress	1.02	1.04	1.27	1.30
	Strain	1.04	1.04	1.85	1.04
Compression	Stress	1.03	1.02	1.24	1.15
	Average for all properties	1.05	1.05	1.37	1.17

Table VII. Ratio of the material properties for the petroleum based resin to the soy-based resin composites.

		UPE/Soy (glass)	VE/Soy (carbon)
Tensile	Modulus	1.10	1.30
	Stress	1.10	1.39
	Strain	0.99	2.03
Compression	Stress	1.12	1.38
	Average for all properties	1.08	1.52

Humidity Aging

Figure 11 shows the weight change of the four materials with humidity aging. The results are expressed as a percentage of the weight of the sample immediately after vacuum. The discontinuities seen at about 1500 hours are the result of a failure of the moisture system of the humidity cabinets, which was corrected after one week. The surge in humidity after the correction caused a jump in the moisture absorption values.

The weight changes follow a log-normal curve, $y = m \ln(x) + b$. Table VIII shows the values of the slope, intercept, and correlation coefficient, r^2, for the tensile samples. Correlation coefficients for all the samples are above 0.9, indicating a good fit of the data to the equation. For both the glass-reinforced samples and the carbon-reinforced, the soy-based resins show a steeper slope of moisture absorption than do the petroleum-based samples, indicating that the soy composites are more sensitive to moisture. The carbon fiber-reinforced composites also show a steeper slope than the glass fiber-reinforced composites. In the earlier report, it was concluded that the fiber-matrix interface for the carbon composites may need improvement, which would contribute to increased moisture absorption by the composite.

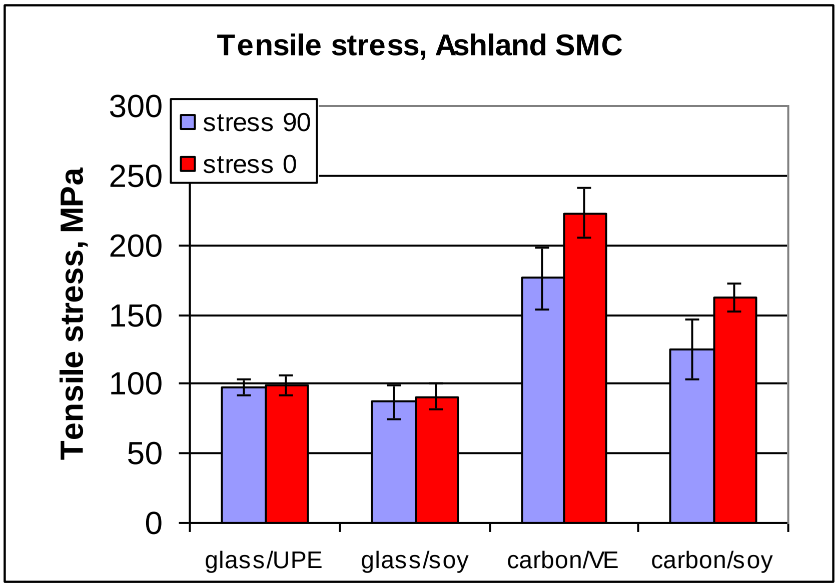

Figure 7. Comparison of tensile stress for petroleum-based and soy-based SMC materials. Values are given for both 0º and 90º orientation. Error bars are standard deviation.

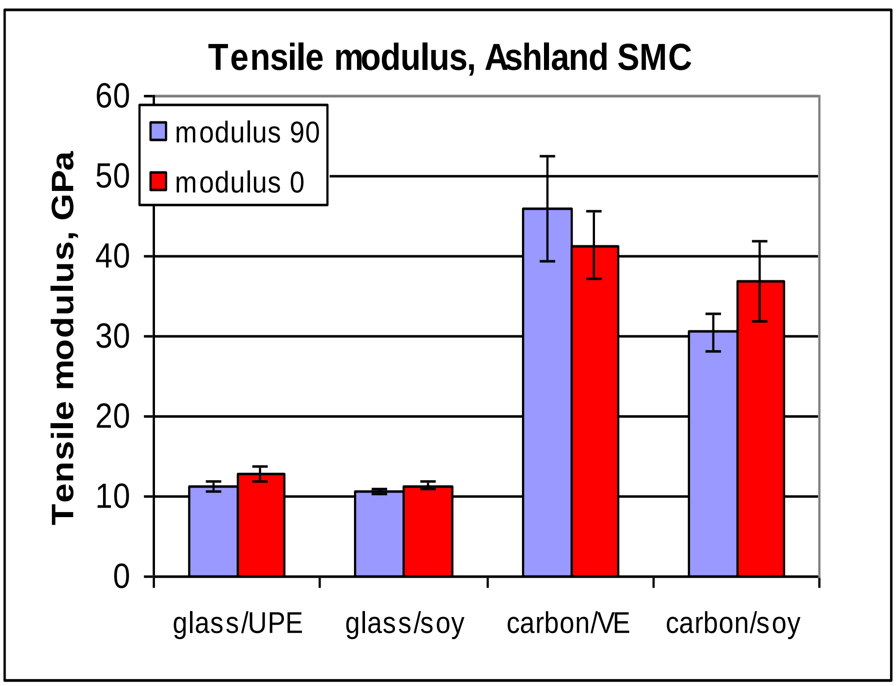

Figure 8. Comparison of tensile modulus for petroleum-based and soy-based SMC materials. Values are given for both 0º and 90º orientation. Error bars are standard deviations.

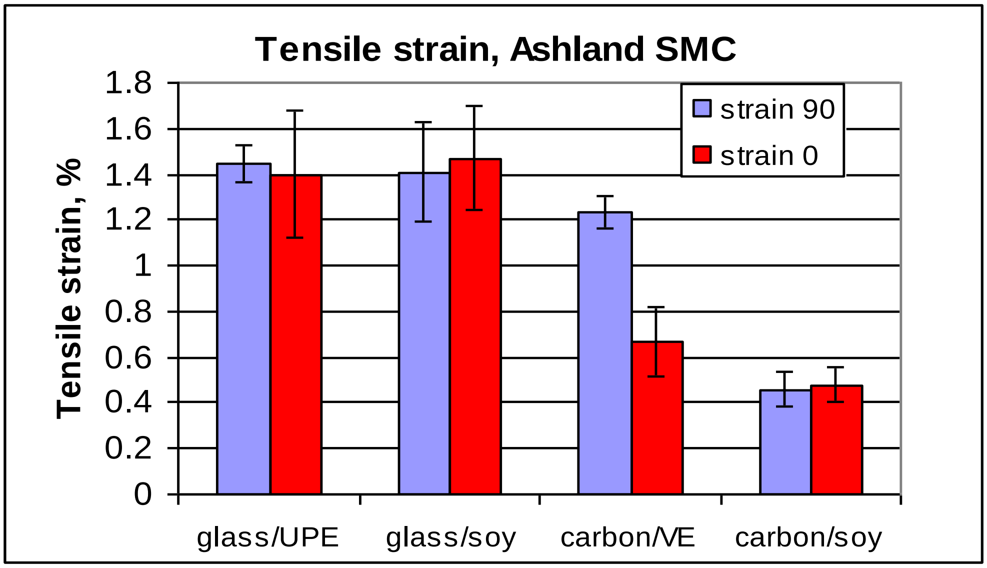

Figure 9. Comparison of tensile strain for petroleum-based and soy-based SMC materials. Values are given for both 0º and 90º orientation. Error bars are standard deviations.

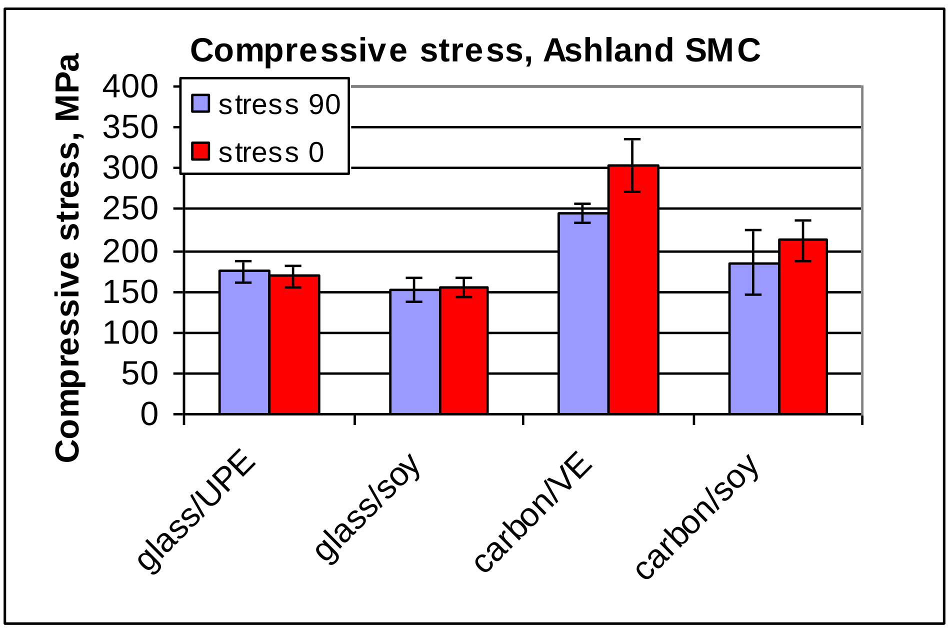

Figure 10. Comparison of compressive stress for petroleum-based and soy-based SMC materials. Values are given for both 0º and 90º orientation. Error bars are standard deviations.

Table VIII. Equation parameters for log-normal curves of moisture absorption for the tensile samples. Intercept is evaluated at 1 hour, since these are log plots.

	Slope (m)	Intercept (b)	Correlation coefficient (r^2)
glass/UPE	0.07%	0.25%	0.971
glass/soy	0.12	0.14	0.940
carbon/VE	0.30	-0.90	0.979
carbon/soy	0.51	-1.22	0.995

Figure 12 shows the comparison of the weight gained at 2000 hours for the four sets of SMC. For the glass-reinforced materials, the soy-based samples gain about 30% more moisture than the petroleum-based resin. However, for the carbon-reinforced composites the gain for the soy-based resin is almost double for both the compression and tension samples. As seen in the equation coefficients in Table VIII, the carbon-reinforced composites both show more moisture absorption than do the glass-reinforced.

Mechanical Properties after Humidity Aging

The AR mechanical properties given in Tables IV and V were measured in both the 0º and 90º directions, but not on the samples for humidity aging. To conserve material, we measured properties in only one direction in the humidity aging. Since it was not certain that the plaques were controlled for charge direction when molded, we elected to compare values after humidity aging to the average of the 0º and 90º properties. Table IX gives the tensile and compressive properties after humidity aging, with the coefficients of variation.

As was seen in the AR samples, the soy-based composite values were lower than the petroleum-based. In Table X the ratio of the petroleum-based samples to the soy-based samples for both the AR and the HA samples is shown. For the glass materials, the ratio values range from 0.87 for the compressive strain to 1.27 for the tensile stress. The mean of all the glass HA values is 1.12. Leaving out the compressive modulus and strain, which were not measured for the AR values, the mean is 1.16, compared to 1.08 for the AR samples. It may be expected that the ratio would be higher for the HA samples, as the soy resins absorbed more moisture than the petroleum-based resins, and therefore are more likely to have degraded properties.

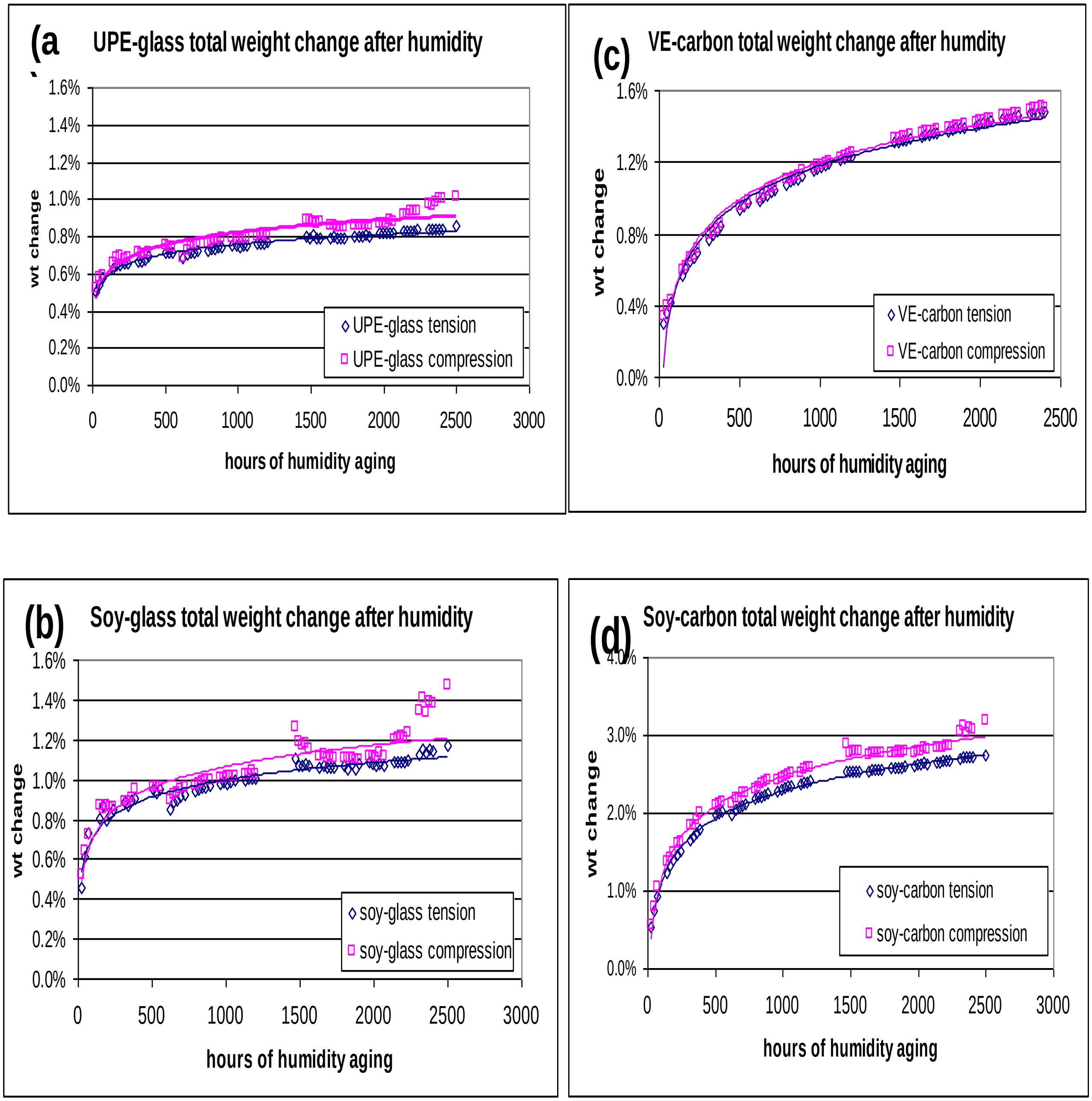

Figure 11. Weight Change with humidity aging, expressed as a percent of initial sample weight, for petroleum-based and soy-based composites. (a) UPE/glass. (b) Soy resin/glass. (c) VE/carbon. (d) Soy resin/carbon.

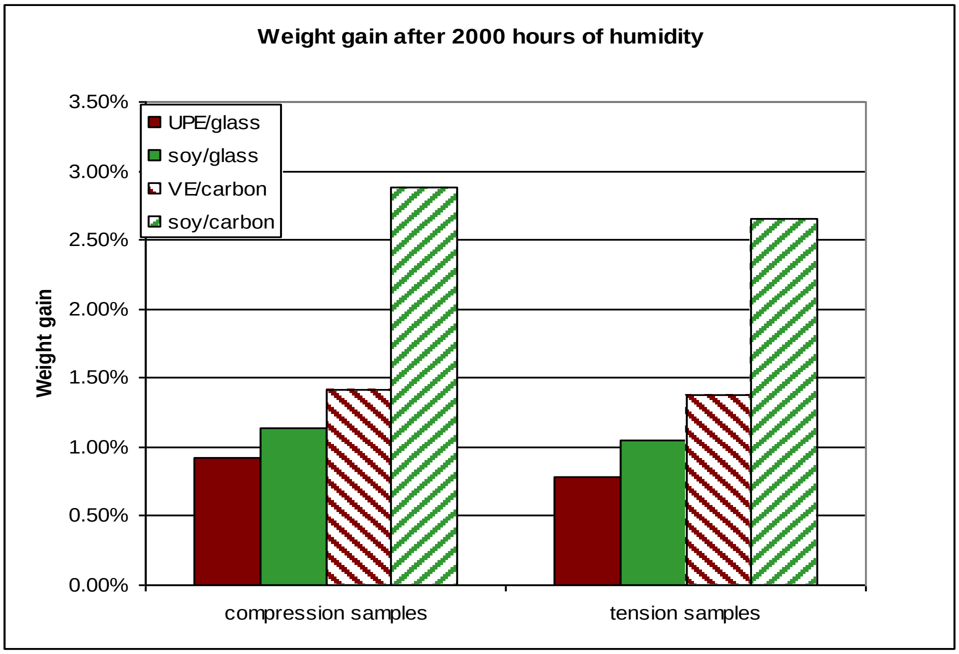

Figure 12. Comparison of weight gain for compression and tension samples at 2000 hours of humidity aging.

Table IX. Tensile and compressive properties for petroleum-based and soy-based SMC after humidity aging. COV values are coefficients of variation, the standard deviation divided by the mean, expressed as percent.

	tension					
	stress (MPa)	**COV**	**modulus (GPa)**	**COV**	**strain (%)**	**COV**
UPE/glass	66.5	7%	11.5	7%	0.89	9%
Soy/glass	51.84	14%	9.5	13%	0.82	14%
VE/carbon	190	30%	32.7	17%	0.63	33%
Soy/carbon	134	15%	25.1	15%	0.54	48%
	compression					
	stress (MPa)	**COV**	**modulus (GPa)**	**COV**	**strain (%)**	**COV**
UPE/glass	121	7%	9.4	13%	1.31	18%
Soy/glass	112	5%	7.7	8%	1.5	10%
VE/carbon	235	18%	34.4	24%	0.7	27%
Soy/carbon	141	21%	24.4	40%	0.68	23%

For the carbon-reinforced samples, however, the petroleum/soy ratio was actually higher for the AR samples, with a mean of 1.52 for the AR, compared to 1.33 for all six HA properties, and 1.39 omitting the compressive modulus and strain, even though the soy resin absorbed about twice as much moisture as the petroleum resin. The largest discrepancy between the AR and HA carbon samples is the tensile strain ratio. For the AR samples this is 2.03, compared to 1.17 for the HA. However, for the carbon compressive stress, the HA value is higher than the AR: 1.67 compared to 1.38. The tensile stress and modulus ratios for the AR and HA are quite similar. The petroleum/soy ratios that are the highest are from the properties that are the most susceptible to the resin degradation, the tensile strain and the compressive stress. The values show that moisture degradation may be an issue for these materials, but these ratios may not be good quantitative measures.

Table X. Ratio of the material properties for the petroleum-based to the soy-based composites.

		As received		After humidity aging	
		UPE/Soy (glass)	**VE/Soy (carbon)**	**UPE/Soy (glass)**	**VE/Soy (carbon)**
Tensile	Stress	1.10	1.39	1.27	1.42
	Modulus	1.10	1.30	1.21	1.30
	Strain	0.99	2.03	1.09	1.17
Compression	Stress	1.12	1.38	1.08	1.67
	Modulus			1.22	1.41
	Strain			0.87	1.03
	Average for all properties	1.08	1.52	1.12	1.33

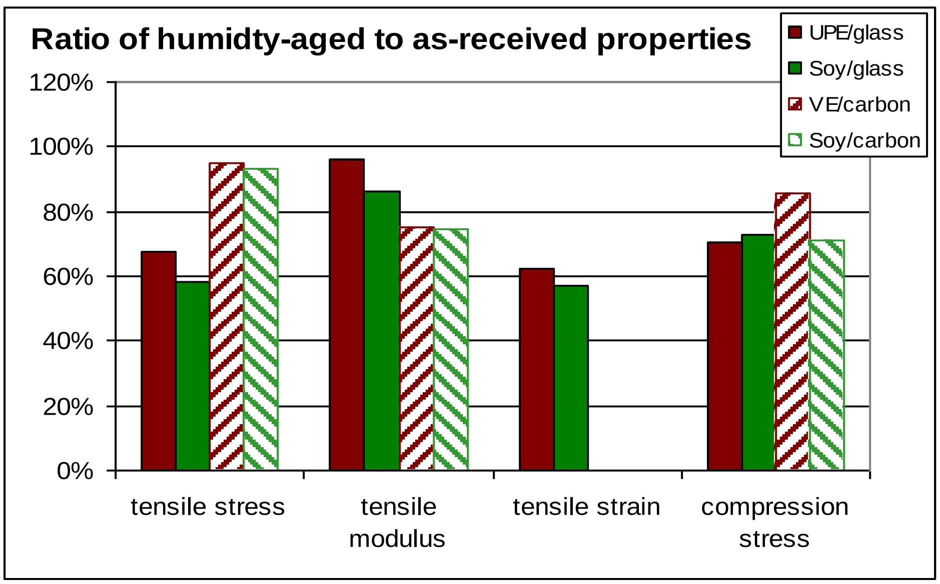

Figure 13. The ratio of the tensile and compressive values for the humidity-aged to as-received materials.

Figure 13 shows the ratio of the HA to the AR values for the soy-based and petroleum-based composites. Tensile strain values are not shown for the carbon-reinforced materials because of the high values of COV for these samples (Table IX). For most of the HA/AR ratios, the soy-based materials perform very similarly to the petroleum-based. The soy-based resins started with lower properties, but lost about the same percentage as the petroleum-based resin. For the glass reinforced materials, the HA/AR ratios for the tensile properties for the soy-based materials are only slightly lower than for the petroleum-based materials, and the compressive stress is not significantly different. Both the soy- and petroleum-based carbon-reinforced composites show less effect of the moisture than do the glass-reinforced materials for the tensile stress, but greater effect for the tensile modulus, and similar effect for the compressive stress. The petroleum-based and soy-based properties are similar for all measures except for the compressive strength, for which the VE/carbon ratio is 0.86, and the soy/carbon is 0.71.

Summary and Conclusions

For the glass-reinforced SMC, the performance of the soy-based material is similar to the petroleum-based SMC in terms of dynamic mechanical analysis (DMA), coefficient of linear thermal expansion, tensile properties and compressive strength. For carbon-reinforced SMC, the thermal properties of the soy-based material are also close to those of the petroleum-based product. However, the mechanical properties of the soy-based material are significantly lower than those of the petroleum-based material. Although the soy-based SMC materials absorb more moisture during humidity aging than do similar petroleum-based materials, the relative tensile and compressive property loss caused by humidity aging was similar for the petroleum-based and soy-based SMC composites. More development is needed by the resin and fiber suppliers to improve the fiber-matrix interface for these materials, particularly for the carbon-reinforced materials.

These materials potentially provide the automotive industry with a method of decoupling composite materials costs from petroleum prices, and of decreasing the lifecycle CO_2 evolution of our vehicles.

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