NMSBA Project Report:

Tests for potential degassing of hazardous organics from alternative recycled insulating materials

Prepared For

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Summary of Conclusions

Upcycle Santa Fe requested technical assistance to study and document the rates and types of off-gassing and emissions produced from recycled insulation materials in comparison to available insulation products on the market. A set of 12 simulated building structures were assembled, each with a different insulating material ranging in insulation type from standard fiberglass batts and straw to a suite of recycled materials produced by Upcycle Santa Fe. The structures, approximately 1 meter by 1 meter by 1 meter cube each were insulated with 10 centimeters of insulating material. Samples were collected from each structure at two different times (morning and midday) on Oct 17, 2016. Analyses were performed at LANL on a Leco Pegasus 4D TOF mass spectrometer with EI at 70 eV. The data were deconvoluted with Leco ChromaTOF software, and the mass fragment patterns were searched against NIST14 Mass spectra database. In only two samples were minor contaminants found and only one of those cases was a building insulated by Upcycle products. LANL also performed measurement of the temperature of the interior and exterior surfaces of all of the buildings in order to make an estimation of the comparative efficacy of the various insulating materials. All upcycle insulating materials provided measureable insulating properties. LANL advises Upcycle Santa Fe that with the techniques used, there are no observable emissions from the recycled insulating materials under the conditions measured and that the materials are effectively insulating to varying degrees.

Assistance Provided

Los Alamos National Laboratory (LANL) provided assistance to Upcycle Santa Fe to investigate the feasibility and safety of employing recycled products produced by Upcycle for use as insulating materials for residential homes. Specifically, LANL agreed to investigate the potential for degassing or off-gassing of potentially hazardous organic emissions and compared these results to emissions from other more standard commercially available insulating materials. LANL employed several state of the art analytical tools which are further described below. In addition LANL made relative comparisons of the efficacy of the different insulating materials that, although not providing a rigorous test of the insulating factor (R value) of Upcycle products, does provide some guidance toward best use and practices as products are further developed and refined.

Experimental Design

In consultations between LANL and Upcycle representatives, it was determined that the best path toward determining emissions from insulating materials was to construct a set of dimensionally identical structures that were each insulated with different insulating materials ranging from Upcycle products through other commercial recycled products to more standard fiberglass and foam insulation. The structures were to be constructed with standard two by fours one meter to a side (Figure 1, left). The interior faces were cover with galvanized wire screening to hold insulation in place and the exterior walls sealed with 1/8 inch particle board while the roofs were covered with corrugated tin, overhanging by 3 inches on all sides. The structures were completely sealed with the exception of a half inch pipe under the lip of the roof to facilitate sample withdrawal and internal temperature measurements (barely visible on upper left corner of structures in Figure 1, right).

Figure 1. View of the internal construction of a test structure (left) partially completed showing framework and partially filled with recycled material as insulation. Shown on right are the final 12 completed structures on the day of sampling.

Samples were collected by filling EPA recommended Tedlar sampling bags (Sigma Aldrich, 2 liter maximum volume) with an oil-less, low flow Gast Diaphragm pump (with inert ethylene propylene diene monomer diaphragm). The sampling protocol was to insert a flexible ¼ inch sampling tube, approximately two feet in length into the central portion of the structure volume, purge the tube volume for 1 minute with the diaphragm pump and then fill the expandable Tedlar bag to ~75% maximum volume. Attached to the end of the ¼ inch sampling tube was a K type thermocouple probe to measure the internal temperature of the structure. An infrared surface temperature scanner was also used to measure the temperature of the exterior surfaces of the structures.

The original sampling plan had called for sampling on multiple days in the summer and fall in order to obtain a significant temperature range for sampling. Due to issues with respect to acquiring construction materials and assembling the structures, they were not available for sampling until early fall. Even with this delay, we were able to sample each structure twice at both a cool and moderately high temperature on a single day.

Analytical Techniques

Analyses for organic trace gas contaminants were performed on two separate analytical tools. First, a cursory examination was made on an OmniStar Model GSD 320 O2, quadrupole mass spectrometer with tungsten filament and continuous secondary electron multiplier (C-SEM) detector. Tedlar sample bags were connected directly to the heated capillary inlet (200 °C) and mass scans were performed from 20-200 amu at 0.5 AMU resolution. Sample scans were compared against background scans by difference analysis.

Second, analyses were performed on a Leco Pegasus 4D Time of Flight (TOF) mass spectrometer. Samples from the Tedlar bags were transferred to 10 ml glass vials with septa on high vacuum line. 1ml of gas samples were taken from the headspace of the 10ml headspace vials which were then incubated at 55C using a Gerstel MPS2 auto sampler with 1ml headspace syringe. The syringes were incubated at 60C, then the headspace gas was injected into the front Gas Chromatographic system (Agilent 6890N). Separation was performed on a30m x 0.32mm

Supel-Q PLOT column (Supelco) and a 5m x 0.25mm Rtx-1ms column (Restek) connected serially. Helium gas was employed as carrier gas with 2ml/min constant flow. The injection temperature was 250C. The oven temperature was maintained at 35C for 5min. and then heated at 10C/min to 250C, which was held for 3min. Detection was performed by Leco Pegasus 4D TOF mass spectrometer with EI at 70 eV. The data were deconvoluted with Leco ChromaTOF software, and the mass fragment patterns were searched against NIST14 Mass spectra database. Peaks observed under the following conditions were rejected:
1) Same peaks as in the Blanks.
2) Compounds containing Si. Because these are most likely from the GC Column bleeding.
3) Similarity value less than 600. 1000 is perfect match of the mass spectrum to the library.

Carbon Dioxide (CO_2), Methane (CH_4) and $^{13}CH_4$ analyses were performed with a Picarro model number G2132i cavity ringdown spectrometer. Tedlar bags were connected directly to the spectrometer inlet and eluted into cavity at ~30 ml/min. Samples were allowed to reach equilibrium concentration (typically less than 3 minutes) and were then recorded manually.

Finally, surface temperature measurements were made with an Everest Interscience Envrio-Therm infrared temperature sensor. Spot measurements were made from a distance of 12″ (~30 cm) yielding a surface area average temperature for approximately 300 square centimeters of surface. Each of the four vertical walls of the structures was measured (East, North, West and South) and were then averaged and are reported in Table 1 as the difference between the internal temperature and the average external temperature.

Gas Sampling Results

Quadrupole Mass Spectrometer (Q-MS) results: The Q-MS analyses in this study were performed purely as a qualitative test for initial results and to guide the analytical protocol for the subsequent Time of Flight analyses. The initial result from the Q-MS yielded no obvious contamination above background for any species between Atomic Mass Units 20 – 200. This indicated that no dilution would be necessary for the TOF analyses and that highest sensitivity would be required.

Time of Flight (TOF) Mass Spectrometer results: As expected from the initial Q-MS results, there was virtually no contamination of organic species in any of the test structures. Only two samples that were collected on the day of sampling yielded minor trace amounts of any organic contaminant above background and one of those samples was an open air background sample (significant controlled burning was taking place in the mountains east of Santa Fe on the day of sampling and may have contributed to that result). The only test structure to record a “hit” was structure 5, with #1 PET plastic insulation. The scan for sample #5.1.1 yielded five hits with similarity greater than 600 with 2-Phenyl-3-methyl-pyrrolo(2,3-b)pyrazine ($C_{13}H_{11}N_3$) yielding the highest score of 640 out of 1000 relative to the NIST library. Given that there were only two hits in the entire sample set, these are not recorded in Table 1.

The CO_2 and CH_4 analyses were performed as a secondary tool to investigate whether there was any biological activity taking place in the test structures since this would be accompanied by CO_2 production in the case of respiration or CH_4 production in the case of methanogensis. Positive results for CO_2 or CH_4 might be indicative of microbial or fungal growth associated with trapped moisture which could confuse interpretation of TOF results. All CO_2 and CH_4 results are within the range of natural abundance (Table 1) and we conclude that no contamination due to biological activity is present.

Structure #	insulating material	Sample #	time	T(interior)	T(S)	T(E)	T(N)	T(W)	T(ext av)	T(diff)	CH_4	$d^{13}CH_4$	CO_2
				˚C	˚C	˚C	˚C	˚C	˚C	˚C	ppbv	permil	ppmv
0	Background air	0.1.1	9:53	NA	NA	NA	NA	NA	NA	NA	1904	-41	413
1	Eco Bricks	1.1.1	10:08	14.9	34	39	20	20	28.2	13.3	1908	-40	417
2	Carton Ecobricks	2.1.1	10:37	22	35	34	18	19	26.5	4.5	1894	-41	411
3	Ubuntu Blox	3.1.1	10:47	18.5	33	34	22	22	27.8	9.3	1875	-40	406
4	mixed plastic bags	4.1.1	10:12	17.1	35	39	23	19	29.1	12.0	1930	-41	412
5	#1 PET plastic	5.1.1	10:42	28.1	34	29	17	16	24.0	-4.1	1881	-40	408
6	Recycled PET #1	6.1.1	10:20	13.6	40	34	26	24	30.9	17.3	1913	-41	412
7	Recycled Denim	7.1.1	10:55	18.4	39	38	25	25	31.8	13.4	1894	-40	408
8	Fiberglass batts	8.1.1	10:26	19.2	35	38	24	21	29.5	10.3	1891	-41	409
9	Rigid fiberglass	9.1.1	10:16	14.4	35	36	20	22	28.0	13.6	1901	-41	411
10	R-Tech insulfoam	10.1.1	10:52	19.7	38	40	24	26	32.0	12.3	1933	-41	413
11	Straw bale	11.1.1	10:59	18.1	42	39	24	25	32.5	14.4	1894	-40	414
12	Control	12.1.1	10:31	30	36	38	27	26	31.9	1.9	1906	-40	410
0	Background air	0.1.2	14:00	26	NA	NA	NA	NA	NA	NA	1941	-41	413
1	Eco Bricks	1.1.2	13:53	30.5	41	25	26	28	30.0	-0.5	1925	-42	413
2	Carton Ecobricks	2.1.2	13:29	28.6	39	20	19	22	25.0	-3.6	1883	-40	410
3	Ubuntu Blox	3.1.2	13:19	27.2	29	14	13	16	18.0	-9.2	NA	NA	NA
4	mixed plastic bags	4.1.2	13:57	31.1	40	25	28	27	30.0	-1.1	1908	40	409
5	#1 PET plastic	5.1.2	13:25	35	35	18	18	23	23.5	-11.5	1909	-40	411
6	Recycled PET #1	6.1.2	13:45	29.1	38	20	23	24	26.3	-2.9	1885	-40	408
7	Recycled Denim	7.1.2	13:10	26.3	31	19	20	19	22.3	-4.1	1915	-41	410
8	Fiberglass batts	8.1.2	13:39	30.3	37	21	23	27	27.0	-3.3	1909	-41	410
9	Rigid fiberglass	9.1.2	13:49	27.2	41	22	24	25	28.0	0.8	1893	-40	409
10	R-Tech insulfoam	10.1.2	13:15	28.2	32	17	15	16	20.0	-8.2	1914	-41	410
11	Straw bale	11.1.2	13:04	25.5	34	17	15	15	20.3	-5.3	1915	-42	413
12	Control	12.1.2	13:34	33.2	33	21	19	19	23.0	-10.2	1912	-41	409

Results of temperature measurements

Given that the Aspect and Spacing of the structures was not rigorously controlled, the incident radiation on each of the test structures was variable and it was not feasible to perform a scientifically rigorous assessment of the thermal resistance (R-value) of the individual materials. Nonetheless, we attempted to make a qualitative assessment of the relative performance of the various insulating materials by taking the difference of the interior temperature and the average external surface temperature of each unit and then comparing this difference among the full set of structures. The results of this test are shown in Figure 2. Perhaps the place to start with interpreting these results is by looking at the control sample, #12. By mid-morning when the first set of samples were taken, the internal temperature of the control unit was nearly equal to the average wall temperature. In comparison all of the other structures with the exception of sample 5 (#1 PET plastic) maintained a significant differential varying from ~5 to 17 degrees Celsius. By the afternoon when the second set of samples was collected, the internal temperature of the control structure was significantly warmer than the wall temperature indicating that incident radiation on the exterior walls was being converted to thermal radiation and transferred to the interior the structure. The insulated structures all exhibited this behavior to some degree (as exhibited by the negative temperature differentials, red bars, in Figure 2) with the exception of Structure #9, Rigid Fiberglass, which actually retained a positive differential. Again, Structure #5 exhibits anomalous behavior with a temperature differential actually greater than that of the control structure. Assuming that each test structure has the same integrity of seal to the outside air, i.e. no leaky cracks, the structures with the smallest differentials in the afternoon (red bars) can be interpreted to be providing the greatest thermal resistance.

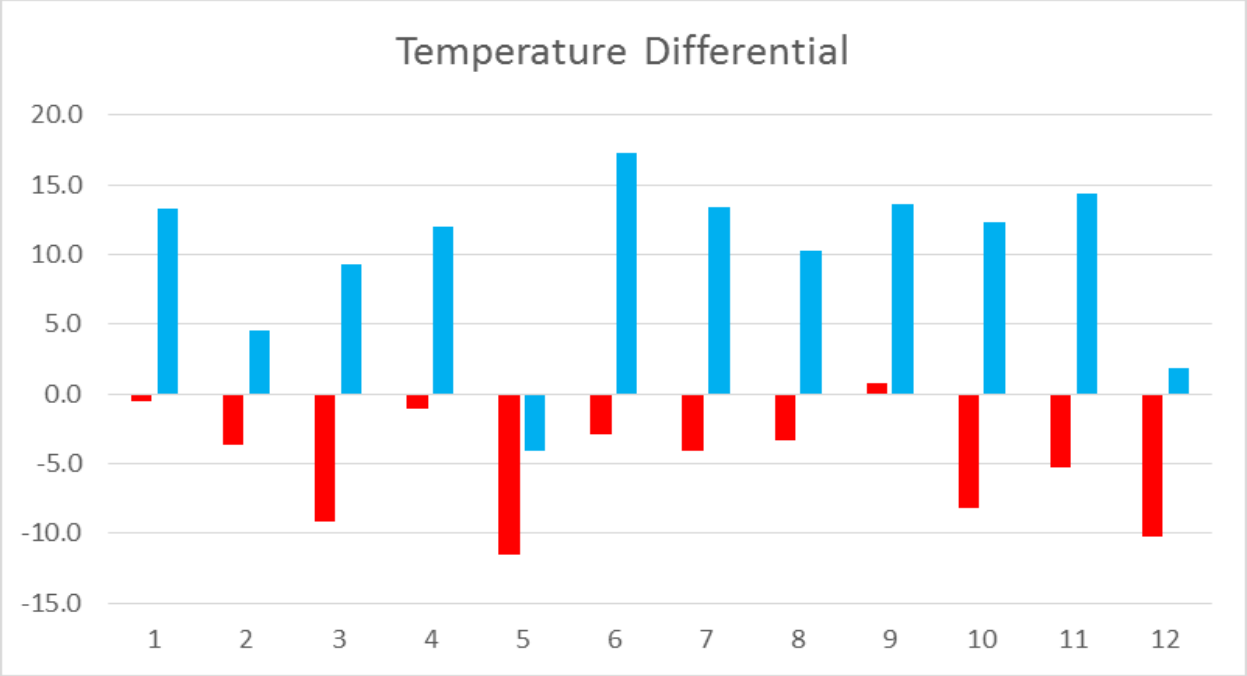

Figure 2. Temperature differential (˚C) between the interior and averaged exterior temperature of the various test structures. Blue bars are from the mid-morning sampling period and red bars are from the afternoon sampling period. Structure numbers correspond to those in Table 1.

Recommendations

LANL advises Upcycle Santa Fe that with the techniques used, there are no observable emissions from the recycled insulating materials under the conditions measured. LANL cannot rule out the possibility that degassing or off-gassing may occur under different conditions than those encountered during the sampling period, e.g. higher temperatures. Additionally, there are other sampling techniques that are more expensive and time consuming than those used for these analyses that may yield different results. Specifically, one more rigorous technique is to cycle test structure air through a molecular sieve or charcoal column for hours in an effort to trap more significant quantities of trace contaminants. This concentrating technique may yield more significant similarities than that observed for sample 5.1.1 for example.

All insulating materials with the exception of #1PET plastic in Structure #5 provided measureable insulating properties of varying degrees. There are of course other more specific technologies as well as certification organizations that are available to quantitatively assess the thermal resistance of insulating materials keeping in mind that quality control is an integral component of reproducible results for a given product.

Overall, we see no reason that Upcycle Santa Fe should not proceed with further development of their alternative, recycled insulating products.