RATIONAL DESIGN OF CELLULOSIC FIBER INSULATION MATERIALS

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Abstract

While fiberglass is a useful and inexpensive insulating material, pulmonary maladies and skin irritation associated with its use make it less desirable to work with or have around in homes and building with children. As previously encountered with asbestos insulation, these issues have created an impetus to actively seek alternatives to fiberglass home insulation. Recently we studied relative R values (thermal resistance) of different nonwoven vegetable fiber composites in a radiation cube with the heat source located at the center. These values per inch thickness of material are the following: cotton 3.8; fiberglass 2.2, jute 4.0; and kenaf 4.4. In addition to these results, in this paper we will discuss new home insulation design criteria, novel sucrose-based adhesives for making flexible non-woven composites adhered to dissimilar substances, and the nature of nonwoven vegetable fiber materials as they affect heat transfer.

Mission Statement

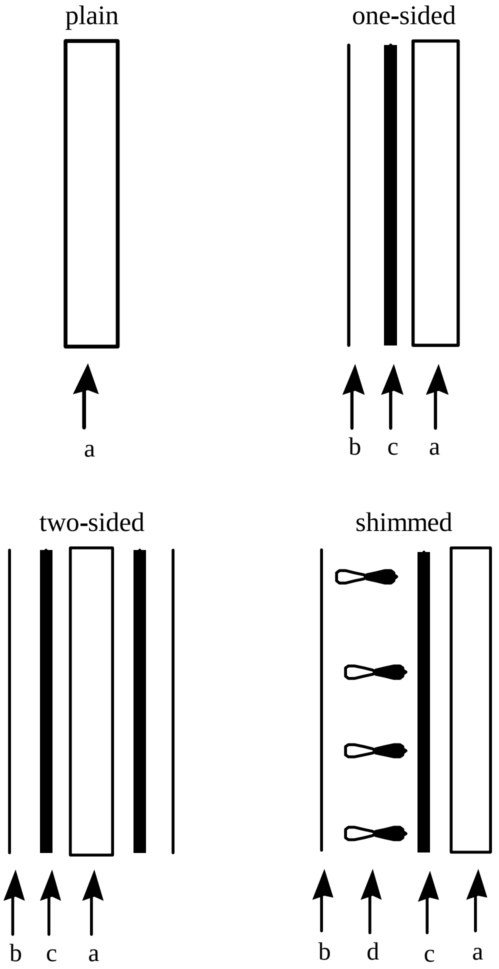
To serve emerging social and industrial needs for low cost, efficient, safe, and environmentally benign materials from agricultural commodities. To create new business opportunities for agricultural commodity groups.

Purpose of This Talk

1 To explain the design of new composite industrial insulation materials by bonding vegetable-fibers to dissimilar surfaces.
2 To demonstrate the development and use of non-toxic and flexible sucrose-derived adhesives for home and industrial use.
3 To discuss the thermal properties of cotton, kenaf, jute, and hemp nonwoven composites as they pertain to the development of architectural insulation.

Composite Arrangements

Thermal resistance and rates of heat transfer will be discussed for the following composite designs and compared with R-19, 6.25 inch batt of fiberglass insulation.



a = vegetable fiber nonwoven fabric
b = aluminum foil
c = flexible, non-toxic, and non-mutagenic sucrose-based epoxy adhesive
d = shim

Figure 1. Four types of composite assemblies tested.

Outline

1. Preparation and structural characterization of sucrose-based epoxies.
2. Comparative testing of adhesives for cytotoxicity and mutagenicity by the Maron Ames tests.
3. Establishing the curing and dynamic mechanical characteristics of adhesives.
4. Characterization of adhesive bond strength by aluminum lap shear tests (ASTM D1002-94).
5. Selection of adhesive formulation for composite assembly.
6. Nonwoven fabrics from vegetable materials.
7. Flame retardant treatment.

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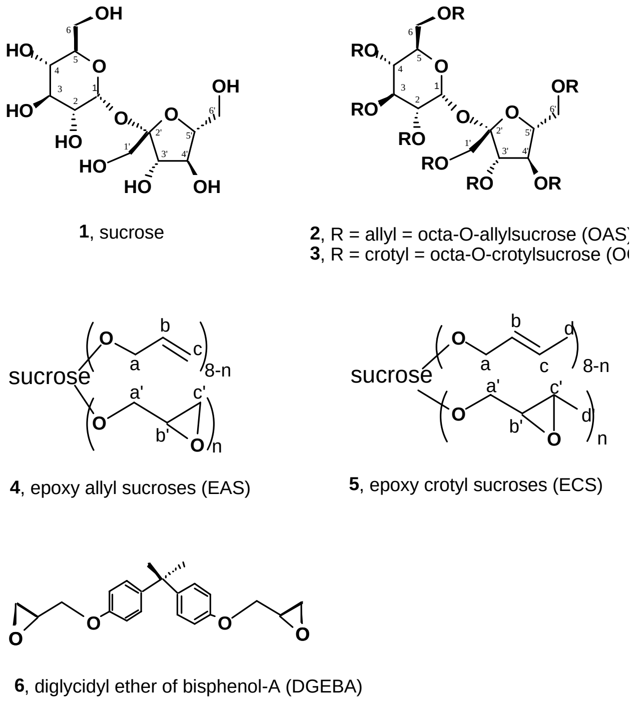
8. Composite assemblies.
9. Heat transfer properties.
10. Rates of heat transfer.
11. Summary and conclusions.



1, sucrose

2, R = allyl = octa-O-allylsucrose (OAS

3, R = crotyl = octa-O-crotylsucrose (O

4, epoxy allyl sucroses (EAS)

5, epoxy crotyl sucroses (ECS)

6, diglycidyl ether of bisphenol-A (DGEBA)

Figure 2. Sucrose- & Bisphenol-A-Based Epoxy Monomers

Modified Maron Ames Test

This test assesses the mutagenicity of chemicals, with or without metabolism of activating enzymes [from a crude sub-cellular fraction, S-9, of rat liver, a rich source of membrane-bound enzyme activity including the membrane-bound oxidizing factor (MFO)] in a range of specialty selected mutant strains of *Salmonella Typhimiurium* bacteria.

Samples:

1. Epoxy Allyl Sucroses-3.2 (EAS-3.2),
2. Epoxy Crotyl Sucroses-7.3 (ECS-7.3), and
3. Diglycidylether of bisphenol-A (DGEBA).

Dilution Factors:

Samples (100 µL) were dissolved in DMSO (900 µL) = 10^1 dilution. Serial 100 fold dilutions were made with DMSO to obtain samples with dilution factors of 10^{-1}, 10^{-3}, 10^{-5}, and 10^{-7}.

Salmonella Typhimiurium strains:

1. TA-98 and
2. TA-100.

Controls:

1. Histidine/biotin requirement (unaltered bacteria),
2. Crystal violet test (membrane permeability), and
3. Ampicillin resistance (integrity of genetic markers).

Assays were performed by Professor Douglas L. Parks Department of Food Science, Louisiana State University, Baton Rouge, LA 70803.

Table 2. Aluminum Lap Shear Tests (ASTM D1002-94).

Epoxy / Curing Agent Formulation	**Break Load (lbs.) [s.dev.]**	**Break Stress (PSI) [s. dev.]**	**Break Stress (MPa) [s. dev.]**
Epoxy Allyl Sucroses-3.2 (EAS-3.2)			
DETA	481	939	6.48
(13 samples)	[54.6]	[103.1]	[0.71]
UR-2142	610	1203	8.30
(20 samples)	[73.8]	[136.1]	[0.94]
UR-2355	371	731	5.04
(20 samples)	[47.5]	[94.0]	[0.65]
Diglycidyl Ether of Bisphenol-A (DGEBA)			
DETA	522	1030	7.10
(29 samples)	[92.5]	[182.8]	[1.26]
UR-2142	764	1507	10.40
(15 samples)	[185.4]	[364.1]	[2.51]
UR-2355	667	1318	9.09
(19 samples)	[113.2]	[222.3]	[1.53]

Selection of Adhesive Formulation For Composite Assembly Criteria

1 Needed Non Toxic & Non Mutagenic Epoxy:

- DGEBA is toxic and mutagenic in *Salmonella Typhimiurium* Strain TA-100.
- Also see *C & E News*, March 24, **1997**, p. 37.
- EAS-3.2 and ECS-7.3 appear to show neither cytotoxicity nor mutagenicity in both *Salmonella Typhimiurium* Strains TA-98 and TA-100.

2 Needed Non Toxic Curing Agent (Manuf. MSDS):

- DETA: "highly toxic, sensitizer, and corrosive."
- UR-2142: "eye irritation, skin sensitization, hazardous transportation."
- UR-2355: "eye irritation, skin sensitization, hazardous transportation."

3 Needed Low Peak Curing Temperature Range:

- EAS-3.2 cures between 85 and 98 °C.
- ECS-7.3 cures between 151 and 191 °C.
- DGEBA cures between 94 and 107 °C.

4 Needed Low Glass Transition Temperature (Tg):

- Tg EAS-3.2 between 8 and 30 °C.
- Tg ECS-7.3 between 50 and 104 °C.
- Tg DGEBA between 84 and 121 °C.

5 Needed Low Young's Modulus (Pa) For Flexibility:

- EAS-3.2: 1.6×10^8 to 1.0×10^9.
- ECS-7.3: 1.4×10^9 to 1.9×10^9.
- DGEBA: 1.4×10^9 to 2.1×10^9.

6 Choice: Cure EAS-3.2 with UR-2355.

Table 3. Compositions of Needlepunched Fabrics.

Sample	Vegetable Fiber (% by wt.)	Polyester (% by wt.)	Poly-propylene (% by wt.)	Fabric Weight (oz. / yd^2)
Air Blown				
1	Cotton 35 %	35 %	30 %	77
2	Jute 35 %	35 %	30 %	82
3	Kenaf 35 %	35 %	30 %	83
Carded				
1	Cotton 35 %	35 %	30 %	28.9
2	Jute 35 %	35 %	30 %	31.2
3	Kenaf 35 %	35 %	30 %	30.8
4	Hemp 35 %	35 %	30 %	29.8
5		70 %	30 %	30.0
6	Jute 50 %	0 %	50 %	26.7
Fiberglass				35

Blending and Needlepunching

- Blend in Rondo Feeder-Webber (1h tumbling).
- Prepare air laid batts [l x w x t = (180 x 45 x 10) cm].
- Cut lengthwise and compress by rolling (to keep batt intact).
- Needle punch in Morrison-Berkshire needlepunch loom twice (thickness ~ 1.3 cm).
- Fold fabric to double thickness (2.54 cm or 1 inch).

Heat Transfer

Theory:

- Heat transfer through a solid material is an energy transition caused by a temperature difference (flux).
- Conduction is primary mode of heat transfer in solids.
- Conduction in direction of thickness is given by:

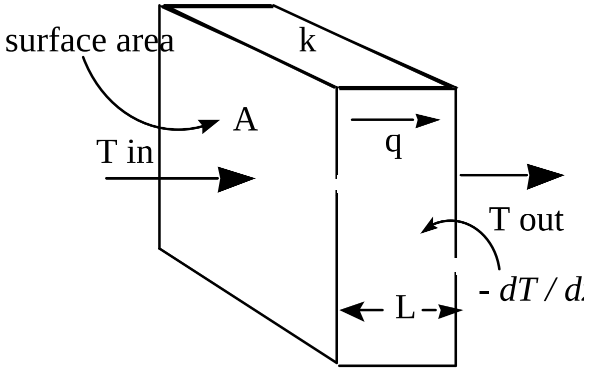
$$q = -kA\,(dT / dx) \tag{1}$$

where:

- q = rate of heat flow, Btu / h
- k = thermal conductivity, Btu / h ft °F
- A = area normal to flow, ft^2
- dT / dx = temperature gradient, °F / ft

Therefore:

- $q \propto$ cross sectional area (A)
- $q \propto$ temperature gradient ($-dT / dx$) through the thickness
- $q \propto k$, and k is the thermal conductivity of the material
- minus sign ⇒ heat flow is in the direction of decreasing temperature.



ΔT = T in minus T out

Integrating [$q = -kA\,(dT / dx)$] along the path of constant heat flow (x - direction), we obtain:

$$q = k\,(A_m / L_m)\,\Delta T \tag{2}$$

where:

- A_m = mean cross sectional area normal to heat flow, ft^2
- L_m = mean thickness of heat flow, ft
- T= overall temperature difference between two sides

R, the thermal resistance of a material (°F h / Btu) is known for common shapes:

- flat wall: $R = L_m / (kA)$;
- insulated pipe: $R = \ln (r_{out} / r_{in}) / 2\pi kL$

Substituting 1 / R in (2) we obtain:

$$q = \Delta T / R \tag{3}$$

Experimentally, T_{in}, T_{out}, and q are established.

Thus, from equations (2 and 3) k and R can be determined.

Summary and Conclusions

1. Thermal conductivities (k) of some known materials [BTU / (h ft°F)]:

- air = 0.016 @ 100 °F
- aluminum = 118 @ 68°F
- asbestos (loosely packed) = 0.093 @ 212 °F
- cork ground = 0.025 @ 90 °F
- cotton = 0.035 @ 100 °F
- epoxy (general purpose) = 0.1 to 0.8 @ 73 °F
- glass wool = 0.022 @ 75 °F
- polyethylene = 0.19 @ 73 °F
- polypropylene = 0.080 @ 73 °F
- PVC = 0.093 @ 86 °F
- wool = 0.027 @ 100 °F

2. Literature shows that thermal resistance (R) is:

- not dependent on the direction of orientation of fibers in batts;
- dependent on thickness of batts; and
- nature of material (thermal conductivity, k).

3. At the INDA Conference (September ’98) we had shown that:

- non-toxic epoxy sucroses were prepared and were able to bind various dissimilar materials;
- the relative thermal resistance R/ inch of cotton, kenaf, jute, and fiberglass to be 3.7, 4.5, 4.0, and 2.2; and
- the vegetable fiber insulation can be rendered resistant to flames by use of readily available materials.

4. Presently we are showing that:

- the relative R per inch values of vegetable fiber batts range from 3.7 to 4.5 per inch regardless their manufacturing process;
- flexible and flame resistant aluminum and vegetable fiber composites can be created using sucrose-based epoxies;
- batts without aluminum, attain steady state heat transfer conditions within 2.0 h;
- batts with aluminum foil facing heat source, attain steady state heat transfer conditions within 1.0 h;
- batts with aluminum away from the heat source, attain steady state heat transfer conditions within 0.5 h;
- batts with aluminum on both sides attain steady state heat transfer conditions within 1.0 h; and
- batts not in direct contact with aluminum, and when the foil faces the heat source, attain steady state heat transfer conditions within 3.5 to 4 h.

References

Sachinvala, N.D. and Litt, M.H. Epoxy Monomers From Sucrose, “**US Patent Nos. 5,571,907** and **5,646,226**. Issued November 5, **1996**. and July 8, **1997**, respectively.

Sachinvala, N.D., Winsor, D.L., Menescal, R.K., Niemczura, W.P., Litt, M.H., and Ganjian, I. Sucrose-based Epoxy Monomers and Their Reactions With Diethylenetriamine. *Journal of Polymer Science, Part A,. Polymer Chemistry Ed.*, **1998**, *36*, 2397-2413.

D. M. Maron and B. N. Ames, *Mutation Res*. **1983**, *113*, 173-215).Table 1. Curing & Mechanical Characteristics Of Sucrose- & Bisphenol-A-Based Epoxies With Diethylenetriamine (Deta), Ur-2142, & Ur-2355

Epoxy	Curing Agent	Epoxy to Curing Agent Mixing Ratios	Peak Cure Temp. (°C)	ΔH cal/g (kcal/mole epoxy)	Young's Mod. (Pa)	Tg (DMA) °C
EAS-3.2	DETA	1 : 0.092 (g)	97.8	84.5 (21.0)	1.0 x 10^{9}	30
EAS-3.2	2142	1 : 0.342 (g)	92.5	51.2 (15.6)	2.9 x 10^{8}	16
EAS-3.2	2355	1 : 0.342 (g)	84.9	51.0 (15.6)	1.6 x 10^{8}	8
ECS-7.3	DETA	1 : 0.442 (g)	151.1	53.7 (7.7)	1.8 x 10^{9}	50
ECS-7.3	2142	1 : 0.625 (g)	191.2	22.8 (4.5)	1.4 x 10^{9}	60
ECS-7.3	2355	1 : 0.625 (g)	168.9	36.1 (7.3)	1.9 x 10^{9}	104
DGEBA	DETA	1 : 0.118 (g)	96.9	126.5 (24.8)	1.4 x 10^{9}	121
DGEBA	2142	1 : 0.440 (g)	107.1	70.9 (17.9)	2.1 x 10^{9}	104
DGEBA	2355	1 : 0.440 (g)	94.0	96.0 (23.9)	1.9 x 10^{9}	84

Table 4. Flame Retardant Treatment Of Nonwoven Fabrics.

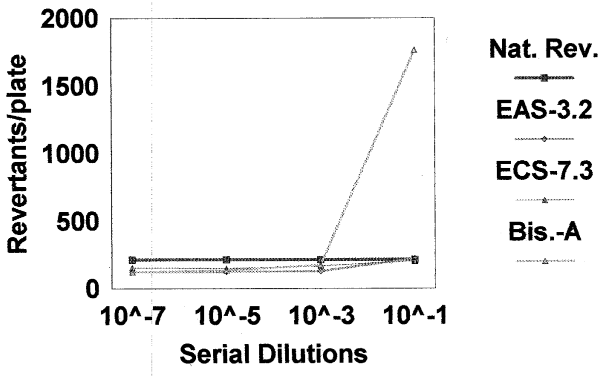
Material	Dry wt. (g)	Wet wt. after spin cycle (g)	% Wet pickup	Wt. after oven drying (g)	% Dry pickup	Results of 12 sec. flame test
Cotton	232	531	128	256	**10.3**	0/4 burned **No glow after burnout**
Jute	239	426	78	249	**4.2**	0/4 burned **No glow after burnout**
Kenaf	220	338	54	231	**5.8**	0/4 burned **No glow after burnout**

Table 5. Heat Transfer Properties Of Composite Assemblies

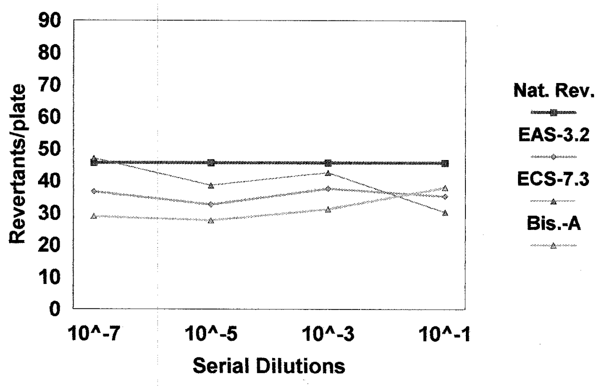
Sample	Tin (°F)	Tout (°F)	T (°F)	Q (Btu/ h)	Thick. (in)	R/inch (°F h/Btu)
1) NO ALUMINUM FOIL						
a) Air Blown needle punched						
Cotton 35 %	174	85	89	24	1.0	3.7
Kenaf 35 %	176	84	92	16.5	1.25	4.5
Jute 35 %	176	84	92	18.5	1.25	4.0
Fiberglass	172	76	96	7	6.25	2.2
Average time to reach steady state heat transfer ≅ 2.0 h						
b) Carded needle punched						
Cotton 35 %	162	95	67	39	0.38	4.5
Kenaf 35 %	164	95	69	37	0.42	4.4
Jute 35 %	162	92	70	36	0.44	4.4
Hemp 35 %	163	97	66	39.5	0.39	4.3
PE 70 % / PP 30 %	162	94	68	35.5	0.48	4.0
Jute 50 % / PP 50 %	162	90	72	34	0.47	4.5
2) ALUMINUM FOIL FACING HEAT SOURCE						
Cotton 35 %	170	87	83	24	1.1	3.1
Kenaf 35 %	170	86	84	20.5	1.1	3.7
Jute 35 %	170	86	84	21.5	1.1	3.6
Average time to reach steady state heat transfer ≅ 1.0 h						
3) ALUMINUM FOIL FACING AWAY FROM HEAT SOURCE						
Cotton 35 %	172	93	79	35	1.1	2.0
Kenaf 35 %	172	92	80	34	1.1	2.1
Jute 35 %	172	90	82	30	1.1	2.5
Average time to reach steady state heat transfer ≅ 0.5 h						
4) ALUMINUM FOIL BOTH SIDES						
Cotton 35 %	165	92	73	29	1.1	2.3
Kenaf 35 %	167	90	77	29	1.1	2.4
Jute 35 %	167	91	76	30	1.1	2.3
Average time to reach steady state heat transfer ≅ 1.0 h						
5) SHIMMED ALUMINUM FACING HEAT SOURCE						
Cotton 35 %	176	80	94	17	1.40	4.0
Kenaf 35 %	176	81	95	15	1.55	4.1
Jute 35 %	176	81	95	13.5	1.55	4.5
Average time to reach steady state heat transfer ≅ 3.5 h						

Fiberglass was commercial, 6.25 inch thick, R-19 insulation purchased from Home Depot.

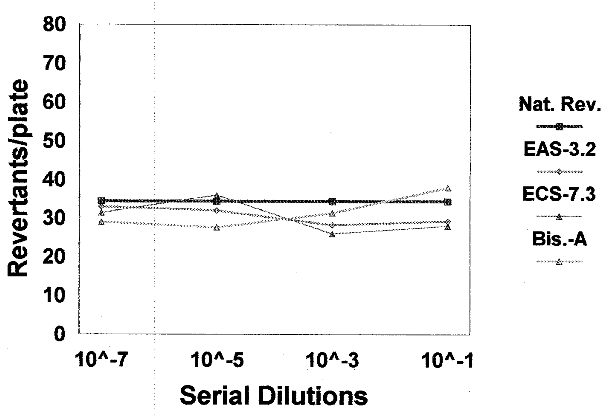
Aluminum foil was 1 mil (0.001 inch) thick, thermal conductivity at 68 °F = 118 BTU / (h °F ft).



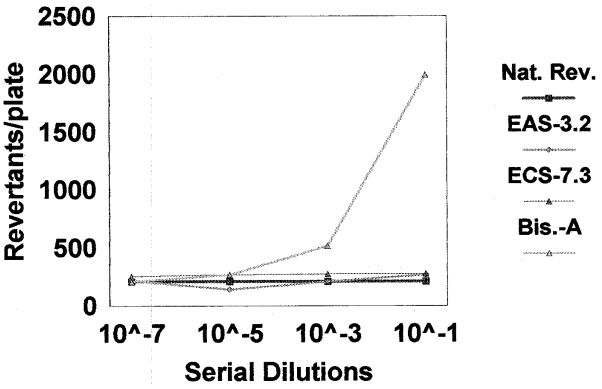
TA-100 with S-9



T-A98 with S-9



TA98 without S-9



TA-100 without S-9

Figure 3. Mutagenic Potential of EAS-3.2, ECS-7.3, and DGEBA with Tester Strain TA-98 and TA-100.