Development of Non-Lead Stabilized PVC Compounds for Insulated Wires and Cables

by Koichi Mizuno * , Hiroshi Hirukawa * , Osamu Kawasaki * , Hideo Noguchi * and Osamu Suzuki *

ABSTRACT With the present trend toward eliminating risks to the environment, electrical appliance and motor vehicle manufacturers have already taken the lead in promoting green procurement and eco-projects, giving preference to the purchase of raw materials having reduced environmental impact. This paper reports on the development of materials using safer Al/ Mg/Ca/Zn-based compounds as stabilizers for the polyvinyl chloride (PVC) used in sheaths for electrical power cables to replace the lead-stabilized compounds conventionally used. It was confirmed that the materials developed exhibited a level of electrical properties and stabilization equivalent to that of lead stabilizers. Power wire products too, were confirmed to be equivalent to products using conventional lead stabilizers in terms of both initial properties and reliability, as shown in long-term tests for resistance to weather and water immersion.

1. INTRODUCTION

At COP3--Session 3 of the Conference of the Parties to the U.N. Framework Convention on Climatic Change--held in Kyoto in December 1997, agreement was reached to reduce emission of carbon dioxide and other greenhouse gases by 5% or more, relative to 1990 levels.

Monitoring of organic substances--paricularly heavy metals--is being similarly strengthened, and the OECD is continuing its risk-reduction activities with respect to five pilot substances: lead, mercury, cadmium, bromine-based flame retarding agents and methylene chloride.

Influenced by this trend toward eliminating risks to the environment, manufacturers of electrical appliances and motor vehicles are taking the lead in adopting green procurement policies that promote the purchase of raw materials that impose a lower environmental burden, and actively pursuing eco-projects. Environmental concerns obviously impinge on the field of power cables too, and measures are being considered to use lead-free and halogenfree materials for PVC sheaths for motor vehicle wire harnesses, wiring for electrical appliances, and the like. Under these circumstances it will be possible, even in the area of PVC insulation for power cables, to replace the lead stabilizers used heretofore with compounds that offer even greater safety.

2. REGULATIONS GOVERNING LEAD

Table 1 shows the various international and Japanese regulations relating to lead compounds. The only specific international regulations are those under the Basle Treaty, and the European Association of Consumer Electronics Manufacturers, as a policy of voluntary restraint, have listed those purchased substances that impact the environment and have established standards for content. When a material purchased appears on this list, the supplier is asked to provide content information. Nearly 50 substances are registered as impacting the environment, including PVC, Cd, hexavalent Cr, Pb and Sb.

Within Japan as well, standards regarding harmful substances have become stricter of late, and restriction on the use of lead and other harmful heavy metals can be expected to be tightened as time goes on.

3. PVC MATERIALS AND THEIR STABILIZERS

The substances most commonly used as stabilizers for polyvinyl chloride are lead-based, and the properties are described below.

3.1 The Function of Stabilizers

Without the addition of a stabilizer, PVC resins begin to degrade at temperatures of 160°C or more, the main reaction being dehydrochlorination. Figure 1 shows the results of measurement of the chlorine in the hydrogen chloride gas produced by the degradation of PVC during heat-

^{* 1&}lt;sup>st</sup> Development Sec., Hiratsuka Lab., R & D Div.

^{*2} Industrial Engineering Sec., Industrial Engineering Dept., Industrial Cables Div.

³³ Factory Management Sec., Insulated Wire Production Dept., Industrial Cables Div.

Table 1 Regulations governing lead

Regulation	Provisions	Mandated levels				
[Internationally] • Basle Treaty	Regulation of import, export, etc. of harmful waste	Pb and Pb compounds: 0.1%				
·EACEM	List of purchased substances impacting the environment	Pb and Pb compounds: 100 ppm				
IEC Guide 109	Guide to design for the environment (DFE)	Elimination of Pb and other harmful substances				
OECD Risk Reduction	Pb, Cd, Hg methylene chloride and bromine-based flame retarding agents	Pb not yet decided; toward voluntary monitoring				
· ISO 14000	Environment management system	14042: Evaluation of environmental impact and influence				
[In Japan] • Law for the Prevention of Atmospheric Pollution	Standard local emission levels for lead	10 mg/m³ or less				
Labor Safety and Public Health Law	Lead concentrations in working environments	0.1 mg/m ³ or less				
Law for the Treatment and Cleaning of Waste Products	Lead dissolution standards for supervisory treatment plants	0.3 mg/l or less				
Sewers Law	Standard levels for discharge into sewers	0.1 mg/l or less				
 Ministry of Health and Welfare Standards for Water Quality 	Lead concentrations in tap water	0.05 mg/l or less				
Law for the Prevention of Water Pollution	Standard levels for purification of underground water	0.01 mg/l or less				
• JIS K 6742	Dissolution of lead from hard PVC water pipes	0.008 mg/l or less				



Figure 1 Heat degradation of PVC

ing. As can be seen, the addition to the resin of a lead stabilizer reduces the amount of hydrogen chloride produced. As heat degradation proceeds, the resin gradually shows brown discoloration, eventually becoming black. At the molecular level, dehydrochlorination causes the formation of double bonds, the cutting of molecular chains, and cross-linking, resulting in a reduction of processability, together with a degradation in mechanical strength and electrical properties. Since PVC is generally processed at temperatures between 160°C (at which degradation begins) and 210°C, it is only through the addition of a stabilizer that it can be molded.

3.2 Lead Stabilizers

At present lead stabilizers are commonly used as PVC stabilizers, and this is particularly true of power cable applications, where lead is used in all but a few specialized areas. The compounds used include white lead, tribasic lead sulfate, dibasic lead phthalate, dibasic lead stearate,

Table 2 Formulation tested

	Parts in formulation
PVC resin	100
(ave. degree of polymerization: 1000)	
Plasticizer (DOP)	50
Calcium carbonate	30
Paraffin wax	0.2
Stabilizer	4

lead stearate and so on. Lead stabilizers are characterized by outstanding thermal stability, low cost, outstanding electrical properties, and by the fact that the lead chloride produced during the stabilization process does not promote dehydrochlorination¹, and there is a need for nonlead stabilizers that can take their place.

4. TARGETS FOR DEVELOPMENT OF NON-LEAD STABILIZERS

In addition to lead stabilizers, there are known to be a number of stabilizers composed of a plurality of metallic soaps, such as Cd/Ba, Ba/Zn, Ca/Zn, and so on. It is further known that organotin stabilizers, and specifically mercaptide, have an extremely high stabilizing effect.

The selection of metallic compounds as non-lead stabilizers was based on the fact that their effect on the human body is slight, and that there was thus little likelihood of their becoming subject to regulation in the future. Possible metals are aluminum, potassium, magnesium, calcium, zinc, etc. Accordingly stabilizers made from these metals were combined and a PVC resin with a non-lead stabilizer was developed for use in power wire insulation. The project was targeted at developing a non-lead- stabilized formulation that was equal to or better than leadstabilized PVC in terms of:

- 1. Static and dynamic thermal stability,
- 2. Electrical properties,
- 3. Mechanical properties,
- 4. Tinting, and
- 5. Processability

5. DEVELOPMENT OF PVC USING NON-LEAD STABILIZERS

5.1 Experimental Methodology

Tests were carried out using a basic formulation of soft PVC used in insulation for electrical wires (see Table 2). The lead stabilizers used for comparison were 4 parts each of tribasic lead sulfate ($3PbO\cdot PbSO_4H_2O$) and dibasic lead stearate ($2PbO\cdot Pb(C_{18}H_{35}O_2)_2$).

In terms of sample formulation and test conditions, the tests were conducted fundamentally in accordance with JIS K 6723-1995 (Plasticized polyvinyl chloride compounds), although some changes were made with respect to measurement times and temperatures. Tests for tensile strength after heating, however, were carried out after heating at 100°C for 5, 14 and 21 days, and the thermal stabil-

ity tests using the Congo red method were carried out at 190°C. For the volume resistivity tests, observations of changes in resistivity after heating and immersion in water were also made, in addition to the standard JIS method.

The degree of tinting of the heated samples was measured in terms of the color difference (ΔE) compared to the original samples. Measurements were made using a JP7200F high-grade spectrum color sensor manufactured by Color Technology Systems.

In addition to static thermal stability tests using the Congo red method, dynamic thermal stability tests for evaluating processing stability were carried out using a Labo Plastomill (30C150) made by Toyo Seiki. Test conditions were jacket temperature of 210°C and roller speed of 100 rpm. Dynamic thermal stability as used here signifies the time from the start of the test to the time at which, once Plastomill torque stabilized, torque began to increase due to scorching of the material.

5.2 Improvement in Electrical Properties

Table 3 compares the stabilization effect of lead stabilizers with those of an ordinary Ca/Zn stabilizer and of one type of Al/Mg/Ca/Zn stabilizer. The Ca/Zn stabilizer was markedly inferior to the lead stabilizer in terms of thermal stability, and volume resistivity, measured at both 30 and 60°C, was also lower. It is therefore impossible, whether in

Table 3 Comparison of lead and non-lead stabilizers

	Unit		Type of stabilizer	
Property	Unit	Pb	Ca/Zn	Al/Mg/Ca/Zn
Initial tensile strength Initial elongation Thermal stability at 180°C	MPa % min.	20 290 315	18 270 170	18 290 475
Volume resistivity at 30°C at 60°C	Ω·cm Ω·cm	6.6E+13 9.5E+12	7.0E+12 4.8E+11	2.1E+13 1.0E+12

		Type of stabilizer					
Property	Unit	Pb	Non-Pb A	Non-Pb B	Non-Pb C		
Initial tensile strength Initial elongation	MPa %	17 250	18 250	18 250	17 260		
After heating at 100°C for 5 days Residual tensile strength ratio Residual elongation ratio	% %	104 97	103 98	100 97	100 98		
∆E * (ab)		2.2	3.3	4.6	3.4		
After heating at 100°C for 14 days Residual tensile strength ratio Residual elongation ratio	% %	107 90	106 89	107 89	108 89		
∆E * (ab)		5.4	9.7	11.9	8.6		
After heating at 100°C for 21 days Residual tensile strength ratio Residual elongation ratio	% %	117 80	111 77	112 79	109 84		
∆E * (ab)		9.2	19.0	18.8	14.6		
Thermal stability at 190°C	min.	100	210	245	250		
Volume resistivity at 30°C at 60°C	Ω·cm Ω·cm	7.2×10 ¹³ 3.6×10 ¹²	2.1×10 ¹³ 1.0×10 ¹²	1.1×10 ¹⁴ 1.3×10 ¹³	1.4×10 ¹⁴ 1.2×10 ¹³		
After heating at 100°C for 21 days and immersion in hot water (60°C) for 3 days	Ω·cm	3.2×10 ¹⁴	2.9×10 ¹⁴	2.9×10 ¹⁴	2.6×10 ¹⁴		

Table 4 Improvement in electrical properties

terms of stability during processing or of insulation performance for the wire, to simply substitute it for the lead stabilizer. The Al/Mg/Ca/Zn stabilizer, on the other hand, had outstanding static thermal stability, and approached lead in terms of volume resistivity, so that it was decided that it would serve as the basis for development of a non-lead stabilizer for power wire sheaths.

Since the Al/Mg/Ca/Zn stabilizer was slightly inferior to the lead stabilizer in terms of volume resistivity, improvements were effected. Table 4 shows the test results. Nonlead stabilizers B and C represent adjustments in the constituents of stabilizer A that influence electrical properties. The values for initial tensile strength and elongation by tensile tests were substantially the same for all of the stabilizers. Even after heating at 100°C for 21 days, the residual elongation ratios for non-lead stabilizers A through C were substantially the same as for the lead stabilizer, demonstrating ample thermal stability. However the color difference (Δ E), which is an index of tinting, was much greater for the non-lead stabilizers A through C than that for the lead stabilizer.

Thermal stability as measured at 190°C by the Congo red method, which was 100 min for samples containing the lead stabilizer, was more than double for all of the nonlead stabilizers. This confirmed that in static thermal stability tests using the Congo red method, the Al/Mg/Ca/Zn stabilizer was superior.

Comparing results in the volume resistivity tests, nonlead stabilizer A was lower than the lead stabilizer, and was below the standard value of $5 \times 10^{13}\Omega$ -cm at 30° C that is mandated for PVC insulation by JIS K 6723. In contrast, non-lead B and C both had higher volume resistivity than the lead stabilizer. It was also confirmed that even when immersed in water at 60° C for 3 days after the heating test, they maintained virtually the same volume resistivity as the lead stabilizer. The purpose of this test was to investigate the effect of the metal chlorides produced during stabilization on the electrical properties.

As has been shown above, non-lead stabilizers B and C both satisfied the development target of electrical properties equivalent to lead stabilizers, whereas both suffered somewhat more tinting when heated. Thus it was decided to select non-lead stabilizer C, which has the lesser discoloration of the two, and to find the optimum formulation using it.

5.3 Improvement in Dynamic Thermal Stability

It having been established that the non-lead C stabilizer offers outstanding electrical properties plus thermal stability, tests were carried out to determine dynamic thermal stability under actual processing conditions.

These tests showed that while, in terms of static stability, the non-lead C stabilizer outperformed lead stabilizers by a factor of 2.5, it was inferior in dynamic thermal stability tests in which a shear was applied. Accordingly evaluations were carried out on three improved versions of sta-



Figure 2 Dehydrochlorination curves at 240°C

Property	Unit	Type of stabilizer					
Topony	Offic	Pb stabilizer	Non-Pb C	Non-Pb C-1	Non-Pb C-2	Non-Pb C-3	
Dynamic thermal stability at 210°C,100 rpm	min.	28	19	26	30	31	
Initial tensile strength Initial elongation	MPa %	21 297	20 313	21 300	21 300	20 303	
After heating at 135°C for 1 day Residual tensile strength ratio Residual elongation ratio	% %	100 92	103 97	99 98	95 101	105 97	
Degree of discoloration ΔE * (ab)		6.7	11.7	14.7	15.6	16.5	
After heating at 135°C for 2 days Residual tensile strength ratio Residual elongation ratio	% %	104 87	103 81	102 81	100 94	102 90	
Degree of discoloration $\Delta E *$ (ab)		12.5	21	32.5	33.4	34.7	
After heating at 135°C for 3 days Residual tensile strength ratio Residual elongation ratio	% %	104 71	113 67	111 69	97 77	106 80	
Degree of discoloration $\Delta E * (ab)$		16	37.8	39.1	41.4	43.1	
Volume resistivity at 30°C	Ω·cm	1.80E+14	5.40E+13	4.40E+13	4.30E+13	4.50E+13	

Table 5 Improvement in dynamic thermal stability

bilizer C (C-1, C-2 and C-3).

Table 5 shows the results of measurements of dynamic thermal stability. It can be seen that the value for the lead stabilizer, at 28 min, and non-lead C at 19 min were the reverse of those for static thermal stability using the Congo red method. But non-lead stabilizers C-1 through C-3 showed improved dynamic thermal stability, with the value for C-2 somewhat better than that for the lead stabilizer. Yet for C-2 more than C-1, and even more for C-3, improvements in dynamic thermal stability tended to be accompanied by greater discoloration (ΔE). The expression $\Delta E^*(ab)$ in Table 5 represents the difference in color from the original, so that a smaller numerical value indicates a lower degree of discoloration. It was judged that non-lead C-2 offered the optimum balance.

6. COMPARISON OF HCI UPTAKE

A non-lead stabilized PVC material was successfully developed, having dynamic thermal stability approaching that of lead-stabilized materials, as determined in sheet property tests and a Labo Plastomill. Here we compare actual HCl uptake with that of lead stabilizer.

6.1 Measurement Methodology

A material containing the stabilizer was placed in a glass container, heated to 240°C, and the amount of chlorine produced was measured in an atmosphere with a nitrogen gas flow of 50 ml/min. The HCl produced by PVC degradation was trapped in water in the form of chloride ions, the concentration of which was measured by an ion

	11-21	Oleandard	Type of s	stabilizer
	Unit	Standard	Pb	Non-Pb C-2
Electrical properties Withstand voltage 1.5 kV	_	Should withstand	0	0
Spark (7500 V x 0.15 sec)	_	Should withstand	0	0
Breakdown voltage	kV	_	29	30
Dielectric resistivity at 20°C (measured at 25°C)	MΩ∙km	50<	7180	1379
Properties of insulation Initial tensile strength Initial elongation	MPa %	10< 100<	25 330	23 320
After heating at 100°C for 7 days Residual tensile strength ratio Residual elongation ratio	% %	(5 days) 85< 80<	93 95	97 100
Oil resistance at 70°C for 4 hrs Residual tensile strength ratio Residual elongation ratio	% %	(tubular) 85< 80<	87 86	93 89
Heating while wound at 120°C for 1 hr	_	Should not crack	0	0
Low-temperature winding (-10°C by JIS)	_	Should not crack	-40°C>	-40°C>
Cold-resistance by JIS	°C	_	-22	-22
Heat shrinkage factor at 100°C for 1 hr	%	3<	0.1	0.1

Table 6	Properties of insulated wire (IV 2 mm ²)

Table 7 Properties of insulated cable (XLPE-insulated with PVC sheath, 3-core x 8 mm²)

		Unit	Standard	Type of s	stabilizer
		Unit	Standard	Pb	Non-Pb C-2
Elec	trical properties Withstand voltage 1.5 kV	_	Should withstand	0	0
_	Dielectric resistivity at 20°C (measured at 25°C)	MΩ∙km	2000<	∞	8
Prop	perties of sheath material Initial tensile strength Initial elongation	MPa %	10< 120<	19 340	22 320
	After heating at 100°C for 2 days Residual tensile strength ratio Residual elongation ratio	% %	85< 80<	100 97	99 98
-	Oil resistance at 70°C for 4 hrs Residual tensile strength ratio Residual elongation ratio	% %	80< 60<	96 89	98 91
_	Heating while wound at 120°C for 1 hr	_	Should not crack	0	0
	Cold-resistance by JIS	°C	-15>	-23	-23
	Heat deformation	%	50>	11.3	9.8
	Flame retardancy	_	Should self- extinguish within 60 sec	0	0

 Table 8
 Properties after accelerated exposure tests

Property		Type of stabilizer		
	Unit	Pb	Non-Pb C-2	
Initial tensile strength Initial elongation	MPa %	24.5 335	23 323	
Embrittlement temperature	°C	-22	-22	
Weatherometer for 2000 hr Residual tensile strength ratio Residual elongation ratio	% %	85 90	91 96	
Embrittlement temperature	°C	-8	-7	
Weatherometer for 2500 hr Residual tensile strength ratio Residual elongation ratio	% %	89 90	94 93	
Embrittlement temperature	°C	-8	-6	

 Table 9
 Properties after outdoor exposure tests

Droportu	Unit	Type of stabilizer		
Property	Unit	Pb	Non-Pb C-2	
Initial tensile strength Initial elongation	MPa %	24.5 335	23 323	
After outdoor exposure for 6 months Residual tensile strength ratio Residual elongation ratio	% %	95 93	96 92	
Embrittlement temperature	°C	-17	-22	
Dielectric resistivity	MΩ·km	514	134	
After outdoor exposure for 12 months Residual tensile strength ratio Residual elongation ratio	% %	95 95	99 97	
Embrittlement temperature	°C	-19	-21	
Dielectric resistivity	MΩ·km	715	167	

analyzer (Shimadzu PIA-1000).

The tests were carried out with respect to samples containing the reference lead stabilized material and a material containing non-lead C-2 stabilizer, together with the ordinary Al/Mg/Ca/Zn stabilizer considered in Table 3, which had not been optimized as described above with respect to the formulations considered.

6.2 Measured Results

Figure 2 shows the results. At the elevated temperature of 240°C, dehydrochlorination was already advanced in the ordinary Al/Mg/Ca/Zn stabilizer after 60 min. In terms of thermal stability at 180°C by the Congo red method, it showed results superior to those of the lead stabilizer. It would seem either that there was a sudden production of HCl at the higher temperature, or that it was impossible to adequately suppress the corrosive effect of the ZnCl₂ produced. In contrast it can be seen that non-lead C-2 maintained a dehydrochlorination suppression effect equivalent to that of the lead stabilizer even at the higher temperature of 240°C.



Figure 3 Insulation resistance (IV 2 mm²) after immersion in water at 60°C



Figure 4 Relative dielectric constant (IV 2 mm²) after immersion in water at 60°C



Figure 5 Electrical properties after loading with 1 kV in water

7. EVALUATION OF PROPERTIES OF INSULATED WIRE

It has thus been confirmed that in terms of thermal stability and electrical properties as measured by sheet tests, equivalence with lead stabilizer has been maintained. Next, using actual extrusion facilities, prototype insulated wires were manufactured using PVC materials containing nonlead C-2, and their usage properties were evaluated in comparison to conventional wire insulated in lead-stabilized PVC.

7.1 Manufacture of Prototype Wire

The prototype wires were vinyl-insulated (IV) wire with a conductor size of 2 mm² and an insulation thickness of 0.8 mm, and cross-linked polyethylene-insulated (XLPE) 3-core wire with a conductor size of 8 mm² and a vinyl sheath thickness of 1.5 mm. In both cases, the prototype exhibited a level of extrudability that was equivalent to that of lead-stabilized PVC.

7.2 Evaluation of Initial Wire Properties

Tests were carried out in accordance with JIS C 3307-1993 (600-V polyvinyl chloride insulated wires) and JIS C 3605-1993 (600-V polyethylene insulated cables and 600-V cross-linked polyethylene insulated cables). Results are shown in Tables 6 and 7. In both cases, initial properties satisfied the JIS requirements.

7.3 Evaluation of Long-Term Properties of Insulated Wire

Insulated wire is a product that has a long service life. Wires and cables for electric power applications in particular are often used outdoors for long periods, where they are constantly exposed to sunlight, wind and rain. The long-term properties of the prototype insulated cables were therefore evaluated under conditions emulating this severe environment. The results were as described below.

7.3.1 Tests to Evaluate Weather Resistance

Accelerated exposure tests were carried out using a weatherometer, under conditions set forth in Japan Cable Standards (JCS #391 (1984)). Sunshine exposure times were 2000 and 2500 hours, and the tests carried out were tensile tests and low-temperature embrittlement tests.

Table 8 shows the results. Compared to lead-stabilized material, equivalent levels of both residual tensile elongation ratio and low-temperature embrittlement were maintained after exposure to sunlight for 2500 hours.

In addition, outdoor exposure tests are being carried forward at Furukawa Electric's exposure testing facility, and data has already been obtained for 6 and 12 months. The exposed wire was subjected to tensile strength tests, lowtemperature embrittlement tests and measurement of dielectric resistivity, and the results are shown in Table 9. Since the period of exposure is still short, there has been no major change in either appearance or properties. We will continue to obtain data for 3 years and 5 years.

7.3.2 Hot water immersion tests

Samples were immersed in water at 60°C for six months, and observations were made of changes in dielectric resistivity and dielectric constant. This test simulated longterm use under high-humidity conditions or in an adverse environment exposed to wind and rain, with the objective of confirming the influence on electrical properties. Measurements were made according to JIS C 3005. Results are shown in Figure 3 and 4. Here too it was confirmed that the properties exhibited were equivalent to those of the lead-stabilized material used for comparison.

7.3.3 Immersed Loading Tests

To determine the effect of water immersion when loaded, samples loaded with a voltage of 1 kV were immersed in water for 3 months, and the subsequent effect on electrical properties was investigated. Figure 5 shows results. Long-term changes in dielectric resistivity showed substantially the same downward trend, and levels were such as to pose no problem for actual use.

8. INTRODUCTION OF THE ECO-ACE® PRODUCT LINE

A program to develop a non-lead-stabilized PVC material that can be used as a replacement for lead-stabilized material in insulated wire and power cables in terms of initial and long-term properties has been completed. Thermal stability, electrical properties, etc. are considered to be substantially equivalent to those of lead-based materials.

Furukawa Electric has begun marketing the newly developed non-lead stabilized PVC sheathed wire under the "Eco-Ace" name. It is of course equally applicable to any of the electric power, telecommunications and vinyl cord applications using PVC insulation or sheathing.

9. CONCLUSION

This paper has reported the development of a non-lead stabilized PVC material in an effort to eliminate compounds of lead, a toxic heavy metal, from PVC materials for insulated wire sheathing applications.

In addition to the problem of heavy metals, PVC materials are also beset with problems involving dioxins. At present there are a number of views on the cause and effect regarding PVC and dioxins, and no consensus has been reached³⁾⁻⁵⁾. PVC is 57% chlorine by weight, and the possibility that this serves as a source of chlorine for dioxins cannot be denied, but conversely it also has a major role in safely fixing the chlorine that is a by-product of caustic soda.

PVCs used for electrical wire has a high rate of recovery and recycling, and product life cycles are long. Further, recent technological advances in final processing, making use of PVC as fuel for blast furnaces, raise the possibility of it being considered more positively from the standpoint of overall environmental impact. PVC is being eliminated from beverage containers and the like, but it may well make new advances in suitable applications.

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