

Adhesives market technical tip

Kristalex™ hydrocarbon resins for use in deep freeze packaging applications

Introduction

Many hot melt adhesives used in product assembly and packaging require both heat and cold resistance to maintain a fiber-tearing bond during hot product filling and subsequent refrigeration or frozen storage. Kristalex pure monomer hydrocarbon resins can be used with EVA polymers to produce low-color adhesives with good adhesion at temperatures from –40° to 140°F (–40° to 60°C) with superior low temperature flexibility and viscosities low enough for good performance on high-speed machinery. This technical tip will explain how to formulate using Kristalex resins to produce packaging adhesives suitable for use in deep freeze applications.

Kristalex™ hydrocarbon resins are water-white, highly color stable, polar, low molecular weight thermoplastic materials prepared from purified aromatic monomers. These resins are compatible with a wide variety of oils, waxes, alkyds, plastics, and elastomers, and are useful in formulating low-color adhesives with good low-temperature properties.

- **Kristalex™ 3070 hydrocarbon resin**—The low softening point of this resin makes it useful in systems that require performance at low temperatures such as frozen food packaging and exterior-use sealants. In EVA-based hot melt adhesives, Kristalex 3070 is compatible with EVA grades containing up to 30% vinyl acetate. Kristalex resins can be used with higher vinyl acetate content if higher levels of wax or aliphatic resins are used.

- **Kristalex™ 3085 hydrocarbon resin**—This moderately low softening point resin is also useful in systems that require performance at low temperatures and is compatible with EVA grades containing up to 30% vinyl acetate. Kristalex resins can be used with higher vinyl acetate content if higher levels of wax or aliphatic resins are used.
- **Kristalex™ 3100 hydrocarbon resin**—This higher softening point resin is compatible with EVA grades containing up to 18% vinyl acetate. Kristalex 3100 can be used with higher vinyl acetate content EVAs if higher levels of wax or aliphatic resins are used. Kristalex 3100 complies with many FDA regulations for applications involving direct contact with food. Compliance with a given regulation in a specific situation should be verified prior to use in a food contact application.

EVA-based adhesives are frequently formulated using C5-based tackifiers, such as Eastman's Piccotac™ hydrocarbon resins. The physical properties of Piccotac 8095, an aromatic modified C5 hydrocarbon resin, and Piccotac 1095, an aliphatic C5 hydrocarbon resin, are compared with Kristalex resins in Table 1. The values shown are an average of typical samples and should not be interpreted as product specifications.

Table 1 Typical physical properties

Hydrocarbon resin	Softening point R&B °C	Polarity cloud point DACP ^a °C	Aromaticity cloud point MMAP ^b °C	Molecular weight distribution		
				Mn	Mw	Mz
Kristalex 3070	70	<–50°C	0	650	950	1450
Kristalex 3085	85	<–50°C	1	650	1150	1900
Kristalex 3100	100	<–50°C	5	700	1500	2550
Piccotac 8095	95	38	76	850	2200	5500
Piccotac 1095	95	52	94	800	1700	3500

^aDiacetone alcohol cloud point

^bMixed methylcyclohexane-aniline cloud point. Note: Lower cloud point temperatures indicate a greater degree of polarity or aromaticity. Full explanations of these tests can be found in Eastman publication WA-86A, Spectrum of hydrocarbon resins.

Adhesives market technical tip

Kristalex™ hydrocarbon resins for use in deep freeze packaging applications (Continued)

Technical discussion

The adhesives industry teaches formulators to match the resin and the polymer for maximum compatibility. There are, however, situations where incompatibility can be a desired property of the adhesive formulation. One such situation is when low-temperature performance is needed, as in deep freeze packaging, which requires flexibility and resistance to cracking at low temperatures. This can be obtained by controlling the level of incompatibility and phase separation in the formulation. Several EVA adhesive formulations with Kristalex and Piccotac resins were tested for cloud point, flexibility, viscosity profile, fiber tear from 140° to –40° (60° to –40°), and cohesive and

tensile strength. Temperature resistance was also tested by measuring shear and peel adhesion failure temperatures (SAFT and PAFT). The formulations are shown in Table 2 along with their Gardner color and 350°F viscosity values where it is seen that the use of Kristalex resins produces water-white adhesives. Evatane™ EVAs are random copolymers of ethylene and vinyl acetate. Evatane 28/40 and 28/420 contain 28% vinyl acetate and have melt indices of 40 and 420 g/10 min, respectively.

Table 2 Adhesive formulations

Component	Wt%				
Evatane™ 28/40 ^a	17.5	17.5	17.5	17.5	17.5
Evatane™ 28/420 ^a	17.5	17.5	17.5	17.5	17.5
Kristalex™ 3070 hydrocarbon resin ^b	40.0	—	—	—	—
Kristalex™ 3085 hydrocarbon resin ^b	—	40.0	—	—	—
Kristalex™ 3100 hydrocarbon resin ^b	—	—	40.0	—	—
Piccotac™ 8095 hydrocarbon resin ^b	—	—	—	40.0	—
Piccotac™ 1095 hydrocarbon resin ^b	—	—	—	—	40.0
Piccotac™ 1020 hydrocarbon resin ^b	10.0	10.0	10.0	10.0	10.0
Paraffin wax 68/70	10.0	10.0	10.0	10.0	10.0
Sasolwax™ C-80 ^c	5.0	5.0	5.0	5.0	5.0
Irganox™ 1010 ^d	0.2	0.2	0.2	0.2	0.2
Gardner color	<1	<1	<1	7	5
Viscosity 350°F (176°C), cP	1275	1255	1710	1235	1275

^aArkema, Inc.

^bEastman Chemical Company

^cSasol Limited

^dCiba Specialty Chemicals Holding, Inc.

Adhesives market technical tip

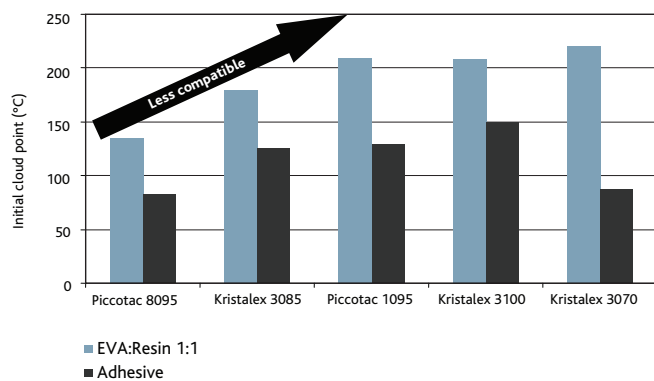
Kristalex™ hydrocarbon resins for use in deep freeze packaging applications *(Continued)*

The compatibility of the adhesive system can be evaluated by measuring the cloud point temperature of the polymer:resin blend and of the formulated adhesive. Cloud point is the temperature at which a blended material begins to separate, causing cloudiness. This is measured by heating the polymer:resin blend or the adhesive until it becomes clear, then allowing it to cool slowly until cloudiness is initially observed. This is the initial cloud point temperature. As the sample cools further, it becomes increasingly cloudy until the thermometer can no longer be seen. This is the full cloud point temperature. The more compatible the components in the test sample, the longer a clear polymer blend is maintained during cooling. Higher compatibility is indicated by a lower cloud point temperature.

Cloud point temperature measurements are shown in Figure 1. The Kristalex resins are less compatible than Piccotac 8095 in the EVA polymers and have higher EVA:resin cloud point temperatures. The compatibility of the waxes with the EVA polymer and with the Kristalex or Piccotac resin contributes to the measured cloud point temperature of the formulated adhesive.

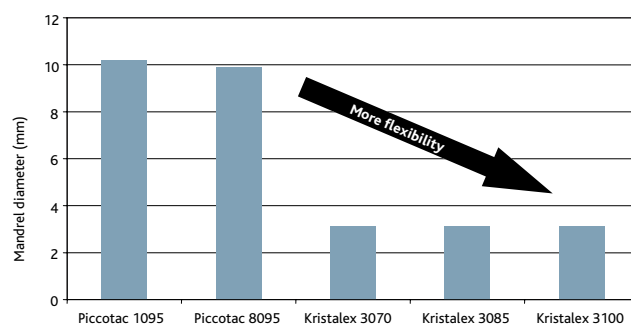
The composition of the phase that initially separates from the adhesive is dependent on the particular tackifier and wax used in the formulation. For these formulations, the cloud point temperatures are lower when compared to the EVA:resin temperatures.

Figure 1 Initial cloud point temperatures of Piccotac and Kristalex resins in EVA



Low temperature flexibility is one property that is improved by using Kristalex resins. As the hot melt adhesive cools, the Kristalex resin separates from the EVA polymer. This multiphase system behaves similarly to a rubber-toughened plastic and can flex without breaking at lower temperatures. The flexibility can be measured by bending the sample around a mandrel with a specified diameter at -20°C . Superior flexibility is indicated by small mandrel diameters. The adhesives formulated with Kristalex resins bent without breaking at -20°C around 3-mm diameter mandrels, while the adhesives with the Piccotac resins broke when bent around 9-mm diameter mandrels.

Figure 2 Adhesive flexibility

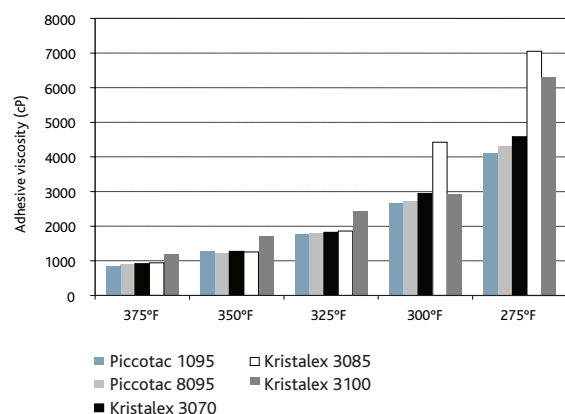


Adhesives market technical tip

Kristalex™ hydrocarbon resins for use in deep freeze packaging applications *(Continued)*

Reduced compatibility can also be used to reduce set time as the Kristalex rapidly separates from the adhesive blend during cooling. While having low viscosity at application temperatures, the EVA-Kristalex adhesives build viscosity with cooling faster than the adhesives formulated with Piccotac 8095 or Piccotac 1095 as illustrated in Figure 3. Set times measured for the EVA-Kristalex adhesives were half the value of the EVA-Picotac 1095 set time. The viscosity increase with cooling can also significantly reduce open times as seen with the Kristalex 3100, but the formulations with Kristalex 3070 or Kristalex 3085 allow a balance of long open time and short set time.

Figure 3 Adhesive viscosity profiles (measured on a Brookfield LV-1 equipped with a thermosel)



The improved adhesion and low temperature flexibility obtained with Kristalex™ hydrocarbon resins is apparent in the corrugated cardboard fiber tear testing shown in Figure 4. As shown, the Piccotac 1095 formulation gives approximately 50% fiber tear on corrugated cardboard at 60°C, and its adhesion decreases with test temperature. Kristalex resin formulations have improved fiber tear from 60° to –40°C. Figure 5 shows this trend is also seen in the tensile strength measurements indicating greater cohesion in the Kristalex formulations.

Figure 4 Corrugated cardboard fiber tear at various temperatures

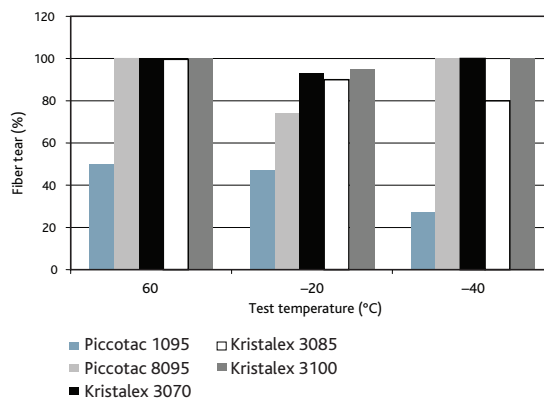
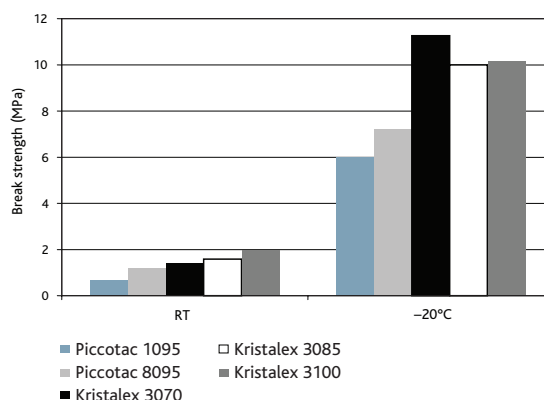


Figure 5 Adhesive tensile properties (test method ASTM D-638 at 1 mm/min)

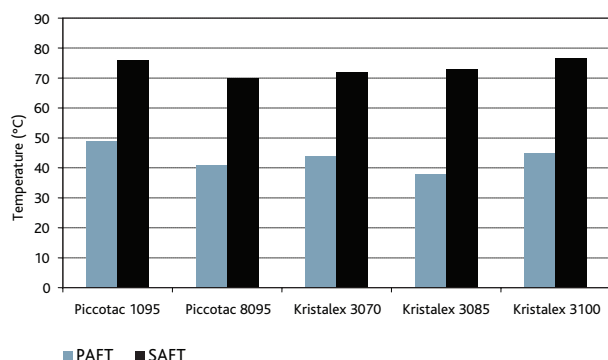


Interestingly, the shear and peel strength of these adhesives is relatively insensitive to differences in the resins used. The shear adhesion failure temperature (SAFT) and peel adhesion failure temperature (PAFT) remain essentially unchanged, preserving high-temperature performance while improving low-temperature performance. These results are shown in Figure 6.

Adhesives market technical tip

Kristalex™ hydrocarbon resins for use in deep freeze packaging applications *(Continued)*

Figure 6 Adhesion failure temperatures
(test method ASTM D-4498 on 40-lb virgin Kraft paper)



The adhesive formulation performance is summarized in Table 3. It shows that Kristalex™ 3070 and Kristalex™ 3085 hydrocarbon resins provide superior performance as deep freeze adhesives. If an open time shorter than 5 seconds can be tolerated, Kristalex 3100 gives excellent performance for deep freeze applications where additional FDA clearances are needed.

Table 3 Adhesive formulation performance

	Target value	Picotac 1095	Picotac 8095	Kristalex 3070	Kristalex 3085	Kristalex 3100
Gardner color	<1	▼	▼	▲	▲	▲
Open time (sec)	≥5	▲	▲	▲	▲	▼
Set time (sec)	≤5	▼	▲	▲	▲	▲
Fiber tear: 60°C	75%	▼	▲	▲	▲	▲
Fiber tear: –20°C	75%	▼	▲	▲	▲	▲
Fiber tear: –40°C	75%	▼	▲	▲	▲	▲
Mandrel flexibility: –20°C	<4 mm	▼	▼	▲	▲	▲

Legend:

▲ = Adhesive met target values.

▼ = Adhesive performance was poor.

Conclusion

Eastman Chemical Company's water-white Kristalex resins can be used with EVA polymers to produce low-color adhesives with improved low-temperature strength and flexibility and with fiber tearing bonds from –40° to 140°F (–40° to 60°C).

For more information on formulation strategies using tackifiers from Eastman Chemical Company, contact us at 1-800-EASTMAN or www.eastman.com/adhesives.



**Eastman Chemical Company
Corporate Headquarters**

P.O. Box 431
Kingsport, TN 37662-5280 U.S.A.
Telephone:
U.S.A. and Canada, 800-EASTMAN (800-327-8626)
Other Locations, (1) 423-229-2000
Fax: (1) 423-229-1193

Eastman Chemical Latin America

9155 South Dadeland Blvd.
Suite 1116
Miami, FL 33156 U.S.A.
Telephone: (1) 305-671-2800
Fax: (1) 305-671-2805

Eastman Chemical B.V.

Fascinatia Boulevard 602-614
2909 VA Capelle aan den IJssel
The Netherlands
Telephone: (31) 10 2402 111
Fax: (31) 10 2402 100

**Eastman (Shanghai) Chemical
Commercial Company, Ltd. Jingan Branch**

1206, CITIC Square
No. 1168 Nanjing Road (W)
Shanghai 200041, P.R. China
Telephone: (86) 21 6120-8700
Fax: (86) 21 5213-5255

Eastman Chemical Japan Ltd.

MetLife Aoyama Building 5F
2-11-16 Minami Aoyama
Minato-ku, Tokyo 107-0062 Japan
Telephone: (81) 3-3475-9510
Fax: (81) 3-3475-9515

Eastman Chemical Asia Pacific Pte. Ltd.

#05-04 Winsland House
3 Killiney Road
Singapore 239519
Telephone: (65) 6831-3100
Fax: (65) 6732-4930

Although the information and recommendations set forth herein are presented in good faith, Eastman Chemical Company makes no representations or warranties as to the completeness or accuracy thereof. You must make your own determination of their suitability and completeness for your own use, for the protection of the environment, and for the health and safety of your employees and purchasers of your products. Nothing contained herein is to be construed as a recommendation to use any product, process, equipment, or formulation in conflict with any patent, and we make no representations or warranties, express or implied, that the use thereof will not infringe any patent. NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE, OR OF ANY OTHER NATURE ARE MADE HEREUNDER WITH RESPECT TO INFORMATION OR THE PRODUCT TO WHICH INFORMATION REFERS AND NOTHING HEREIN WAIVES ANY OF THE SELLER'S CONDITIONS OF SALE.

Material Safety Data Sheets providing safety precautions that should be observed when handling and storing our products are available online or by request. You should obtain and review available material safety information before handling our products. If any materials mentioned are not our products, appropriate industrial hygiene and other safety precautions recommended by their manufacturers should be observed.

© 2013 Eastman Chemical Company. Eastman, Kristalex, and Piccotac are trademarks of Eastman Chemical Company.

www.eastman.com