

Ionomer, acid copolymer, and metallocene polyethylene resins: A comparative assessment of sealant performance

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ABSTRACT: *The performance of 14 commercial grades of metallocene polyethylene (mPE) resins and a traditional Ziegler-Natta catalyzed linear polyethylene (LPE) was compared with a narrow selection of acid copolymer, ionomer, and ethylene-co-vinyl acetate resins. Each of these potential heat seal candidates was characterized for melting behavior, melt strength, blown film processability, heat seal, and hot-tack performance. Although the mPE resins offer enhancements over traditional LPE resins, these polymers lack the overall balance of high melt strength, efficient extrusion processability, versatile heat sealability, resistance to oils, and broad hot-tack performance that is characteristic of ionomer and acid copolymer resins. Metallocene polyethylene resins remain limited in their ability to match the performance of high-pressure.*

KEYWORDS: *Addition polymers, copolymers, ethylene, hydrocarbons, ionomers, olefins, physical properties, polyethylene, polyhydrocarbons, polyolefins, properties, sealability, sealants, tack, thermoplastics.*

The success of multilayer flexible packaging in the food industry has been attributed to the broad use of thermoplastic heat sealants that are capable of providing the highest degree of package integrity, security, and durability (1). The evolu-

tion of the various metallocene catalyst technologies has led to the recent commercial introduction of the metallocene polyethylene (mPE) resins, some of which have been positioned (2, 3) as replacements for ionomer and acid copolymer resins

in heat seal applications. Historically, ionomers and acid copolymers have set the packaging industry's standard with regard to premium and high-performance heat seal applications, and recent studies have concluded (4, 5) that these high-pressure copolymer resins continue to provide the best overall balance of extrusion processability, heat sealability, and hot-tack performance in an initial comparison with the metallocene resins.

A follow-up study was initiated with the acquisition of 14 additional metallocene polyethylene resins that are marketed as potential heat seal candidates for flexible film applications. As listed in **Table I**, each of these polymers was given a prefix designation based upon the catalyst system used in their manufacture. Eight of these were synthesized via the "single-site catalyst" system, while the other six grades were polymerized in the "constrained geometry catalyst" process. All of these are considered to be blown film grades; melt indices ranged from 1.0 to 4.5 g/10 min, while the densities varied from 0.885 to 0.935 g/cm³. The level and type of α -olefin incorporated into the mPE's were determined using carbon 13-nuclear magnetic resonance (¹³C-NMR) analysis. The SSC-designated resins are ethylene copolymers of either butene or hexene or are terpolymers of butene and hexene,

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I. Selected PE resins

Resin code	Grade code	Melt index, g/10 min	Density, g/cm ³	Melt point, °C	Comonomer, wt. %			MWD
					Butene, wt. %	Hexene, wt. %	Octene, wt. %	
SSC-1	2010	1.5	0.924	118	-	4.3	-	2.19
SSC-2	3001	4.5	0.910	103	8.9	-	-	2.04
SSC-3	3006	1.6	0.909	105	1.9	7.5	-	1.90
SSC-4	3025	1.2	0.910	104	6.8	-	-	2.02
SSC-5	3028	1.2	0.901	92	11.2	-	-	1.81
SSC-6	3033	1.2	0.902	95	1.6	10.6	-	2.00
SSC-7	3034	3.2	0.902	95	-	12.0	-	1.98
SSC-8	4011	2.2	0.885	74	16.2	-	-	2.24
CGCT-1	1030	2.5	0.935	126	-	-	3.0	2.37
CGCT-2	1570	1.0	0.915	112	-	-	8.0	2.23
CGCT-3	1840	1.0	0.908	107	-	-	11.0	2.17
CGCT-4	1845	3.5	0.910	104	-	-	11.3	2.30
CGCT-5	1880	1.0	0.902	101	-	-	13.0	2.21
CGCT-6	1140	1.6	0.899	93	-	-	16.0	N/R
LPE-1*	11P3	0.8	0.919	123	8.5	-	-	4.30

*Contains 15% H-P LDPE

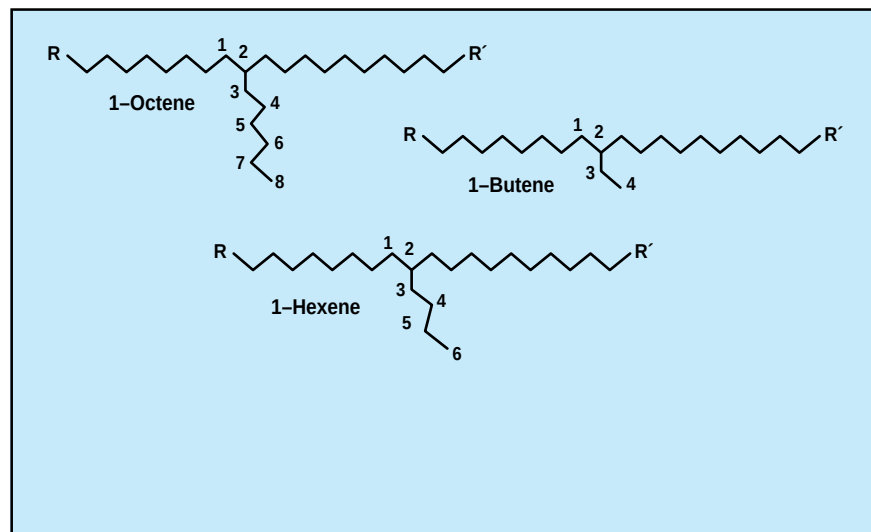
II. Selected high-pressure copolymers

Resin code	Melt Grade code	Comonomer Melt index	point, °C	Type	Content, wt. %	Ion
Ionomer-1	1601	1.3	95	MAA	10	Na
Ionomer-2	1650	1.5	94	MAA	12	Zn
ACR-1	0902	1.1	97	MAA	9	-
ACR-2	1202HC	1.2	95	MAA	12	-
EVA-1	3170	2.5	85	VA	18	-

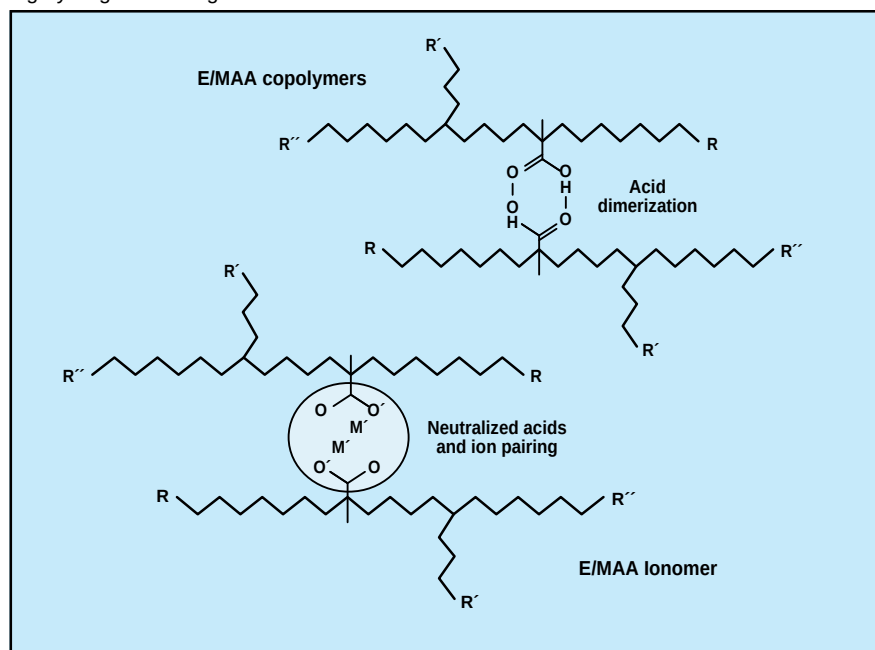
and they range in comonomer content from 4 to 16 wt. %. The CGCT resins are ethylene/octene copolymers ranging from 3 to 16 wt. %. Except for SSC-8, the mPE's appear to meet FDA food contact regulations that affect heat seal layers in food packaging applications. Size exclusion chromatography was used to measure polydispersity; the results indicated that the CGCT resins were slightly broader in molecular weight distribution (2.26) than the SSC resins (2.01).

As a benchmark, a traditional Zeigler-Natta (Z-N) catalyzed linear low-density resin was also included

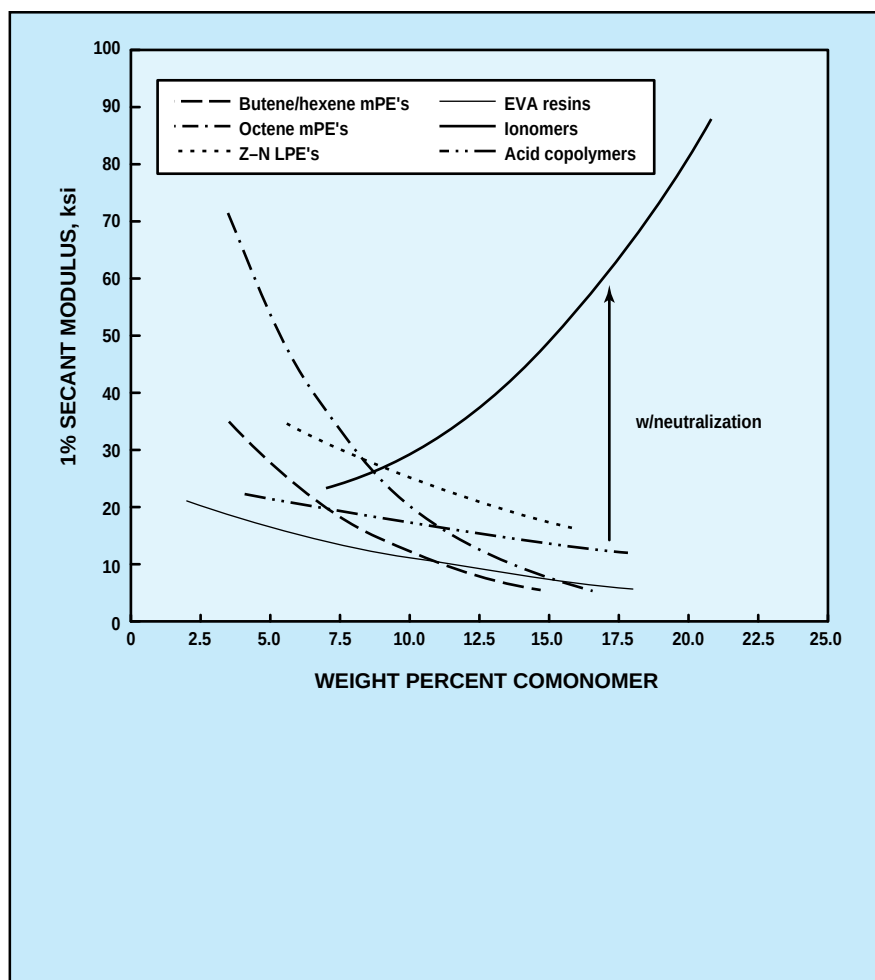
1. Hydrocarbon composition of typical mPE and LPE polymers



2. Chemical composition of ethylene-co-methacrylic acid copolymers and ionomers including hydrogen bonding and ionic intrachain attractions



3. Film stiffness vs. comonomer content



in the evaluation. LPE-1 is an 8.5% butene copolymer that also contains 15 wt. % of an LDPE resin. It is used to gauge the effect of long-chain branching present in the CGCT polymers by approximating the performance of typical linear low-density polyethylene/low-density polyethylene (LLDPE/LDPE) blends.

In comparison, **Table II** lists the selection of the high-pressure, free-radically-polymerized ethylene copolymer resins that were included in the study. Two ionomers, two ethylene-co-methacrylic acid (E/MAA) copolymers, and an 18% Ethylene-co-vinyl acetate (EVA) resin were chosen to represent the copolymer family. Each is commercially available and considered to be a multi-purpose blown film resin. The melt indices of these polymers range from 1.1 to 2.5 g/10 min, while the composition of the acid-based resins range between 9 and 12 wt. % of methacrylic acid. One of the ionomers contains the sodium cation, while the second sample contains zinc. Although these polymers represent a small range of resin design possibilities for the high-pressure technology, they have enabled us to bring the performance of mPE's into perspective.

Polymer chemistry

The attributes of many specialty polyolefins, such as ethylene-propylene-diene monomers (EPDMs), ethylene-propylene rubbers (EPRs) and chlorinated and chlorosulfonated polyethylenes, are derived not just from their molecular structure, but also from their polymer chemistries. Many attributes of acid copolymers and their ionomer counterparts are derived from a polymer chemistry that is unique. **Figure 1** illustrates the typical nonpolar hydrocarbon composition of the metallocene polyethylene resins. In terms of polymer chemistry, there is little difference between the Z-N linear polyethylenes (LPEs) and the metallocene resins, and they offer no additional

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III. Melt strength and extrusion processability

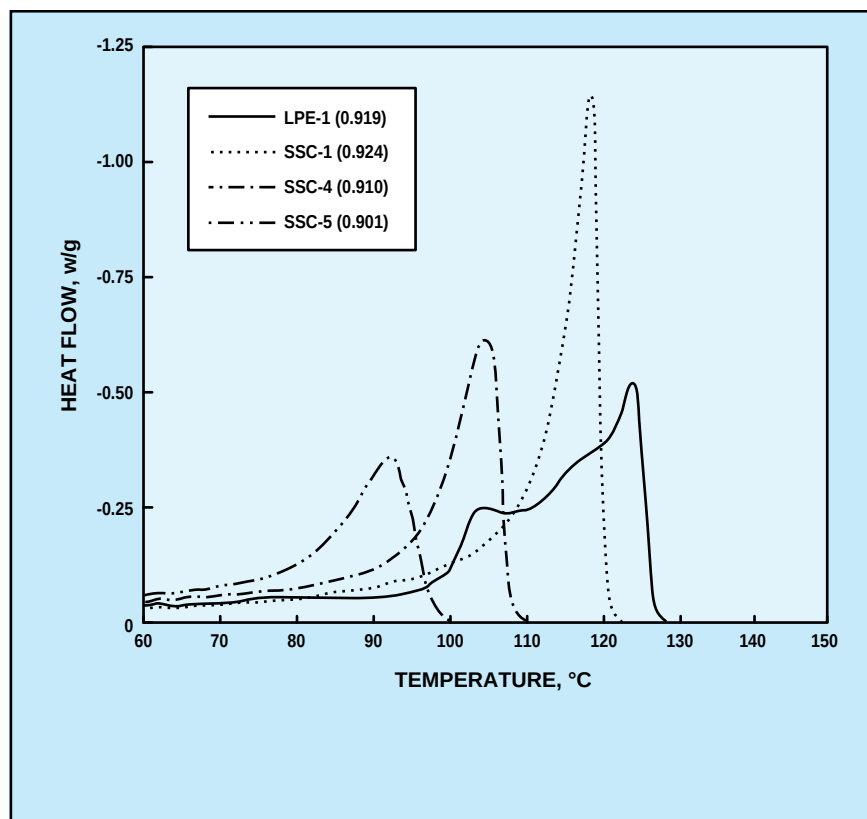
Resin code	Melt index, g/10 min	Melt strength, g	Energy consumption, kW·h/metric ton	Specific throughput, kg/h/100 rpm
SSC-1	1.5	1.7	840	14.1
SSC-2	4.5	0.6	-	-
SSC-3	1.6	1.9	806	15.1
SSC-4	1.2	1.8	885	13.9
SSC-5	1.2	1.9	781	14.3
SSC-6	1.2	2.1	1563	14.4
SSC-7	3.2	1.5	699	16.9
SSC-8	2.2	1.3	-	-
CGCT-1	2.5	2.2	578	13.1
CGCT-2	1.0	3.6	769	14.0
CGCT-3	1.0	4.2	820	14.3
CGCT-4	4.5	1.9	546	16.0
CGCT-5	1.0	2.9	645	15.2
CGCT-6	1.6	2.8	-	-
LPE-1	0.8	4.5	541	12.5
Ionomer-1	1.3	18.8	386	21.4
Ionomer-2	1.5	16.9	437	21.5
Ionomer-3	0.9	21.3	-	-
Ionomer-4	0.9	29.7	-	-
ACR-1	1.1	10.4	420	19.0
ACR-2	1.2	12.5	469	19.5
EVA-1	2.5	6.9	323	24.0

intrachain attractions over their broad-molecular-weight predecessors.

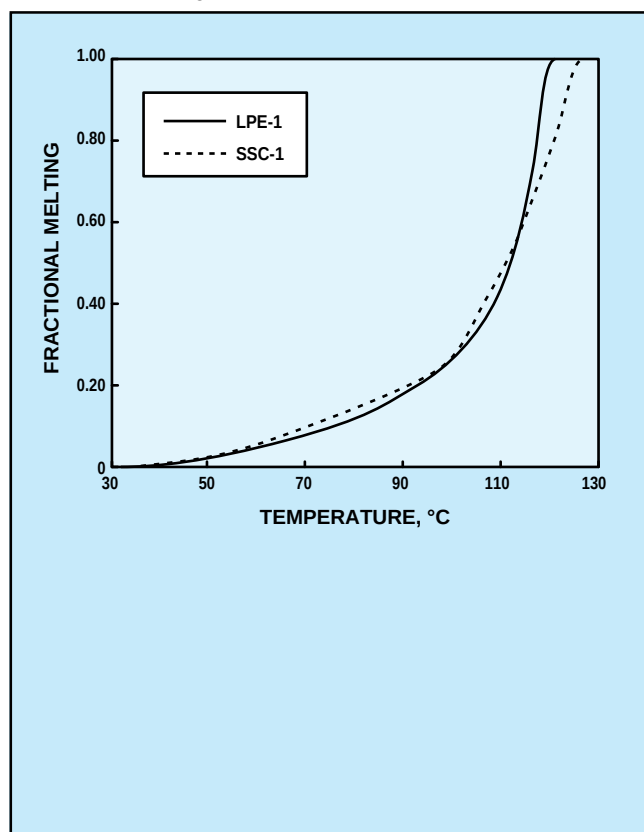
In contrast, **Fig. 2** depicts the chemical compositions of the E/MAA copolymer and the ionomer, its neutralized counterpart. The acid functionality present in both ionomers and acid copolymers provides the opportunity for hydrogen bonding to occur between separate polymer molecules. In the case of ionomers, hydrogen bonding is supplemented by the organization of the neutralized acid/cation species. As these species organize into pairs and multiple clusters, they form dynamic polymer crosslinks that influence polymer properties.

For example, in the copolymerization of ethylene, the introduction of any comonomer serves to disrupt crystallinity, and the polymer trends toward a lower modulus. The impact of neutralizing the acid copolymer resin with a metal cation is evident

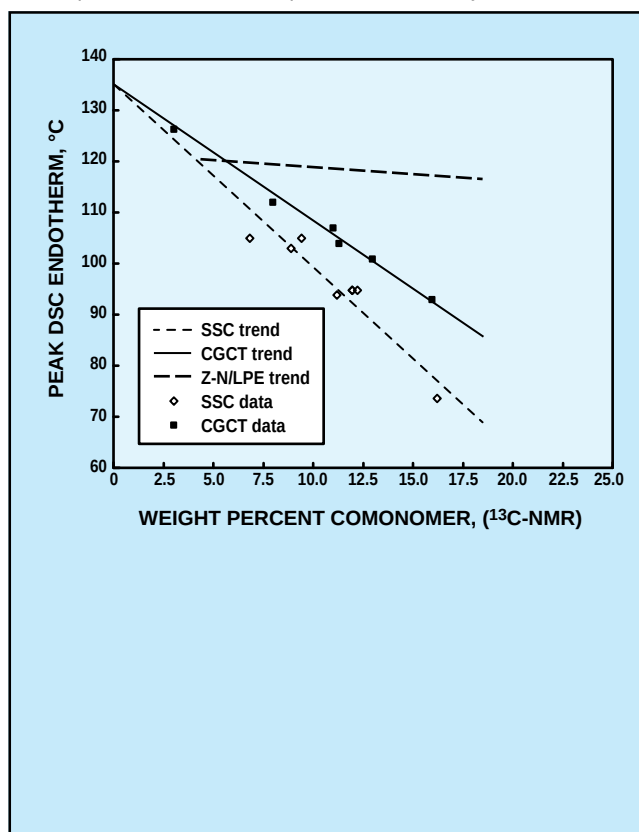
4. DSC melt endotherms Z-N LPE-1 vs. mPE's



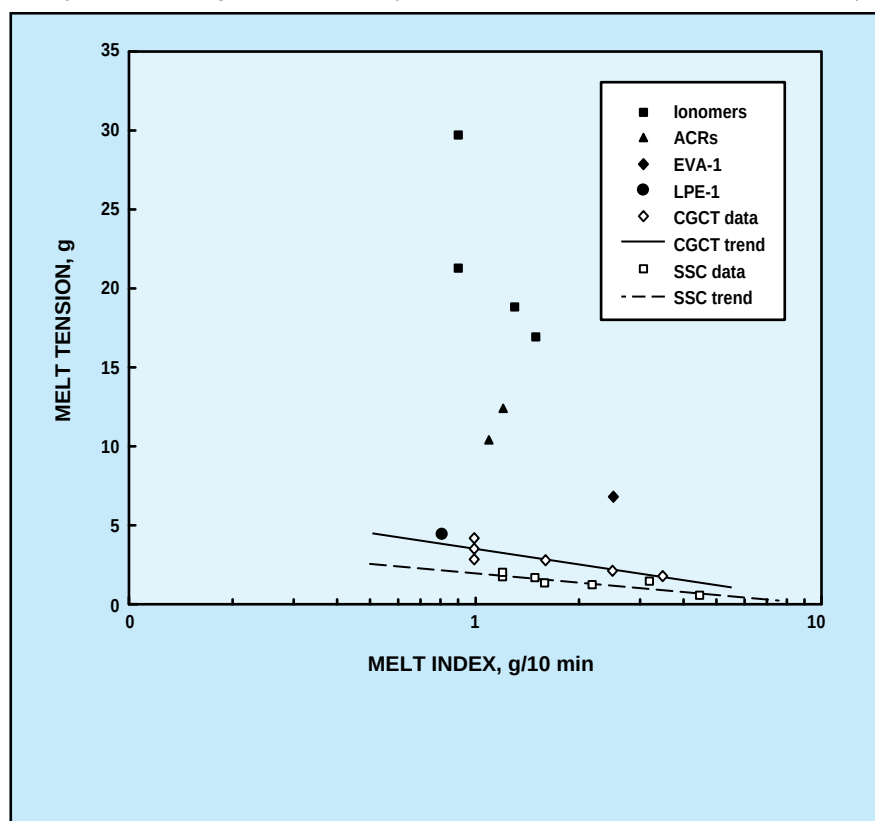
5. Fractional melting of SSC-1 and LPE-1



6. The practical effect of compositional uniformity on melt behavior



7. Polymer melt strength vs. melt index (DuPont method, MI-4 orifice, 0.2 in./min, 190°C)



in **Fig. 3**, as the modulus increases as a function of comonomer content. For ionomers, stiffness can be built back into the resin through control of ion content and degree of neutralization. Likewise, hydrogen bonding and ionic crosslinking help create unique polymer attributes including melt strength, grease and oil resistance, an ability to seal through contamination, and a substantially broader hot-tack operating window.

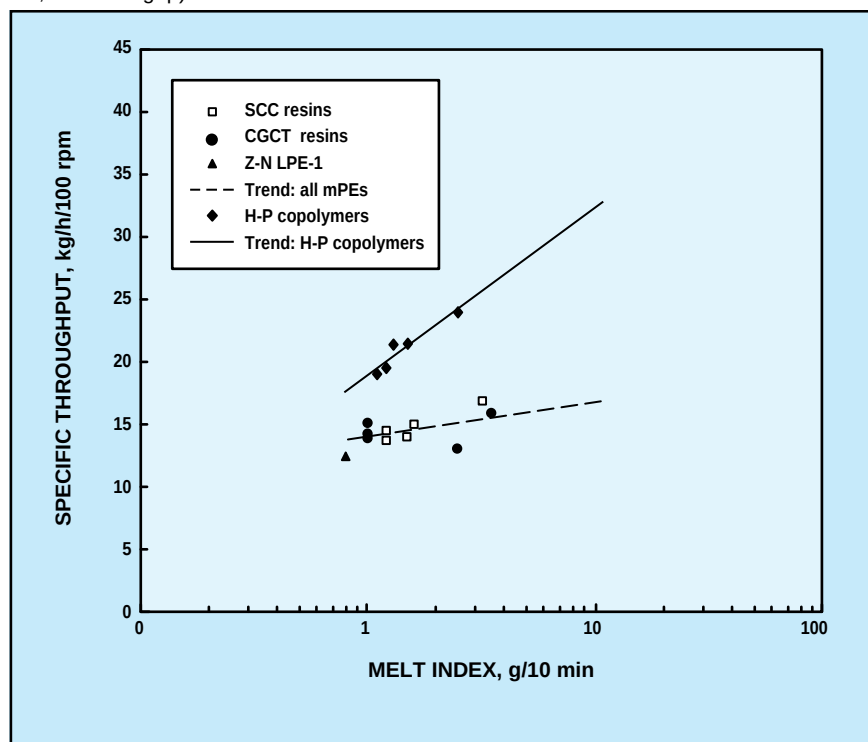
In contrast, metallocene polyethylenes offer improved properties over traditional linear low-density polyethylene (LLDPE) and very-low-density polyethylene (VLDPE) resins due to a tighter control of the molecular weight distribution and compositional homogeneity, and not because of any changes in polymer chemistry. The fact remains that metallocene catalyst technologies are not useful for the copolymerization of ethylene and methacrylic acid.

Unique thermal properties

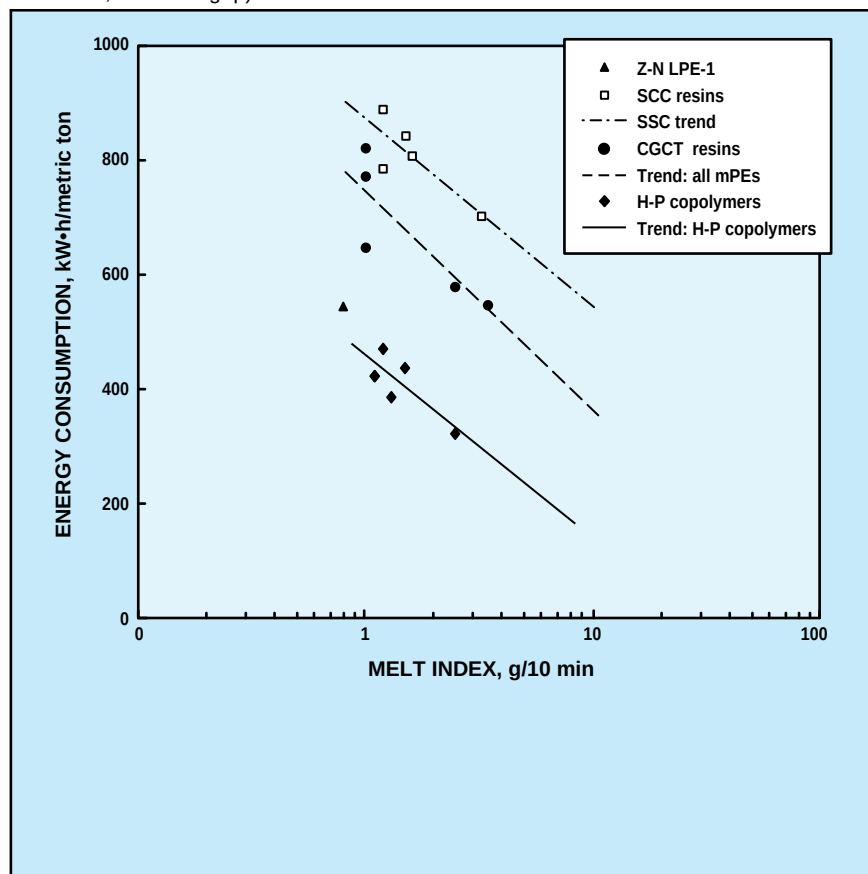
The unique structure/property relationships for the metallocene polyethylene resins were first described by Stehling, *et al.* in 1991 (6) and 1992 (7) and then by Knight and Lai (8) a year later. The much discussed homogeneity of the α -olefin distribution along the polymer backbone results in both lower and sharper melting points. While describing the use of differential scanning calorimetry (DSC) fractionation for the characterization of comonomer distribution in ethylene copolymers, Keating and McCord (9) confirmed that a "single-site metallocene" polyethylene specimen melted in a narrower temperature range compared to a conventionally prepared LPE. Our results also confirmed that the metallocene polymers have an enhanced melting behavior. In **Fig. 4**, the melt endotherms of several mPE's are sharper in comparison to the endotherm of LPE-1. In **Fig. 5**, the fractional melting curve for SSC-1 (0.924 g/cm³) indicates that it reached total melting 5–10°C sooner than did LPE-1.

The practical effect of compositional homogeneity is given in **Fig. 6**, where polymer melt point is given as a function of comonomer composition. The relative flatness of the Z-N/LPE relationship is indicative of the high-molecular-weight, low-comonomer-content fraction that is present in traditionally catalyzed LLDPE and VLDPE resins. In contrast, the metallocene catalyst's ability to produce polymers with compositional uniformity results in a stronger relationship between composition and melt point behavior. The use of lower-molecular-weight α -olefins makes the SSC process appear more efficient at reducing melt point than does the CGCT process.

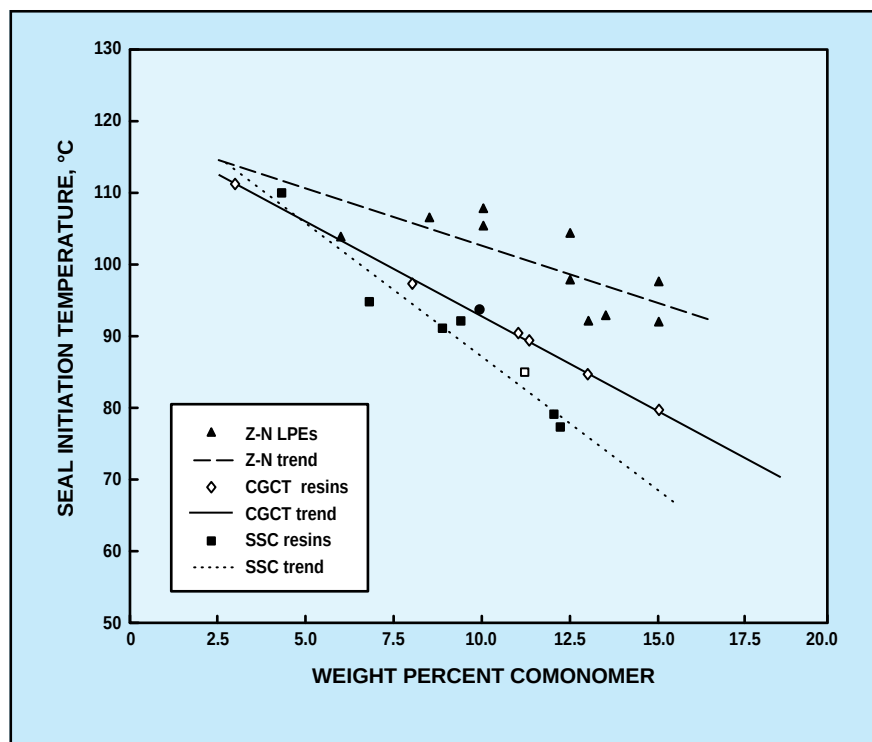
8. Blown film processability—extruder output vs. melt index (40-mm screw, 16:1 L/D, 10-cm die, 0.50-mm gap)



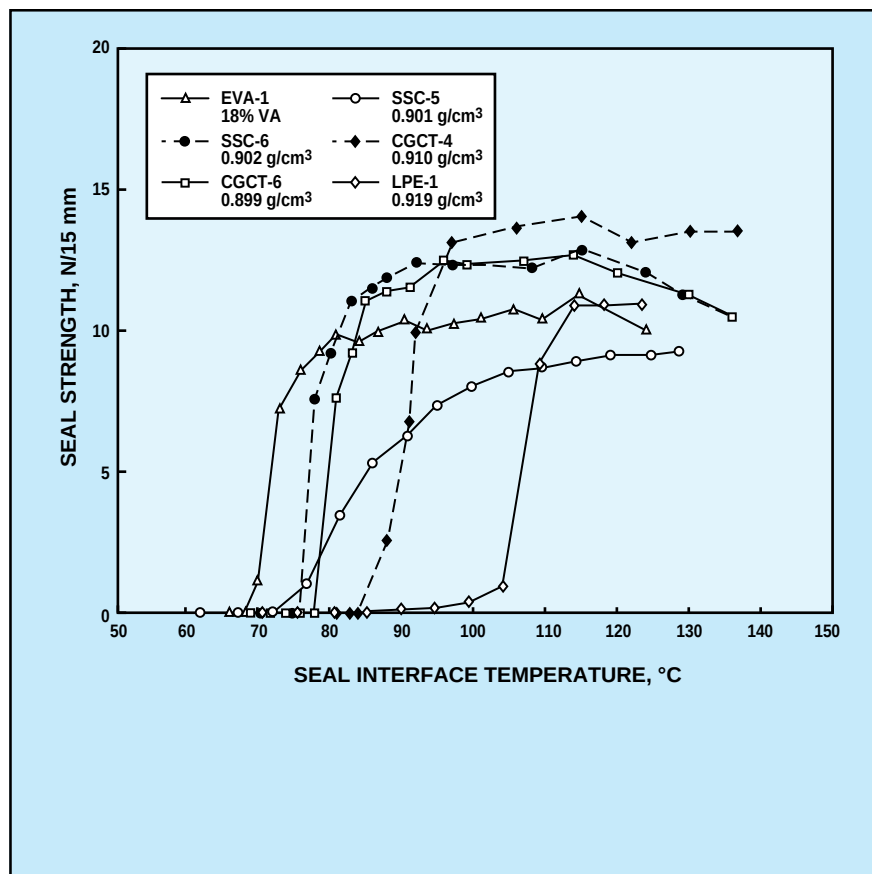
9. Blown film processability—energy consumption vs. melt index (40-mm screw, 16:1 L/D, 10-cm die, 0.50-mm gap)



10. Heat seal initiation temperatures—mPE's vs. Z-N LPEs



11. Low-temperature sealability of mPE's and EVAs



Melt strength and extrusion processability

Melt strength and extrusion processability are two attributes of ionomers and acid copolymers that are important to converters and end users alike. Converters rely upon high-melt-strength polymers to provide bubble stability in blown film and to add balance between neck-in and drawability in extrusion coating and cast extrusion. They also benefit when resins have higher extruder output and require less energy to convert into film. In end-use applications such as thermoforming, the melt strength of ionomers and acid copolymers is needed to eliminate package blow-outs and to ensure that key layers of the film structure are carried evenly into the deep pockets and tight corners of the thermoforming cavity.

Both molecular structure and polymer chemistry influence the melt strength and extrusion processability. Molecular weight, molecular weight distribution, and the degree of long-chain branching affect these properties for polyethylene, while hydrogen bonding and ionic crosslinking add to the character of acid copolymers and ionomers. For the linear polyethylene resins, the absence of long-chain branching is a disadvantage that can be mitigated by blending with highly branched polymers (10, 11) such as LDPE and EVAs.

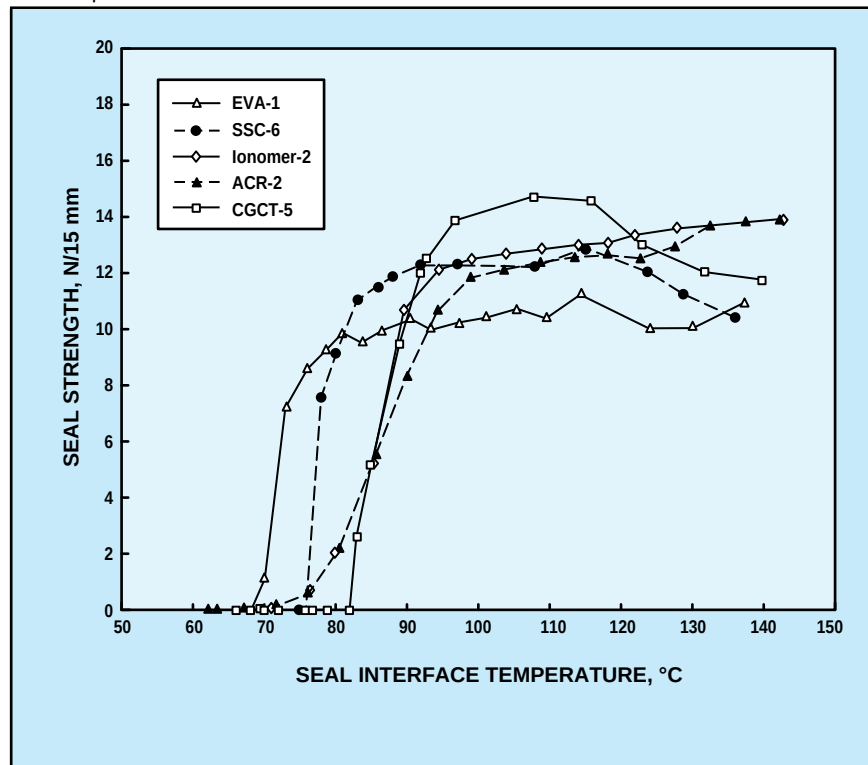
The relative melt strength of different polymers can be characterized by extruding molten polymer out of a capillary die at a constant shear rate. Simultaneously, a take-up reel is used to draw the polymer into a thin strand that is run across a microtensometer. This tensiometer measures the melt tension of the polymer as the draw rate is steadily increased to the point of breakage. The maximum tension applied just prior to breaking is recorded as the melt strength. The results of our melt strength evaluation are listed

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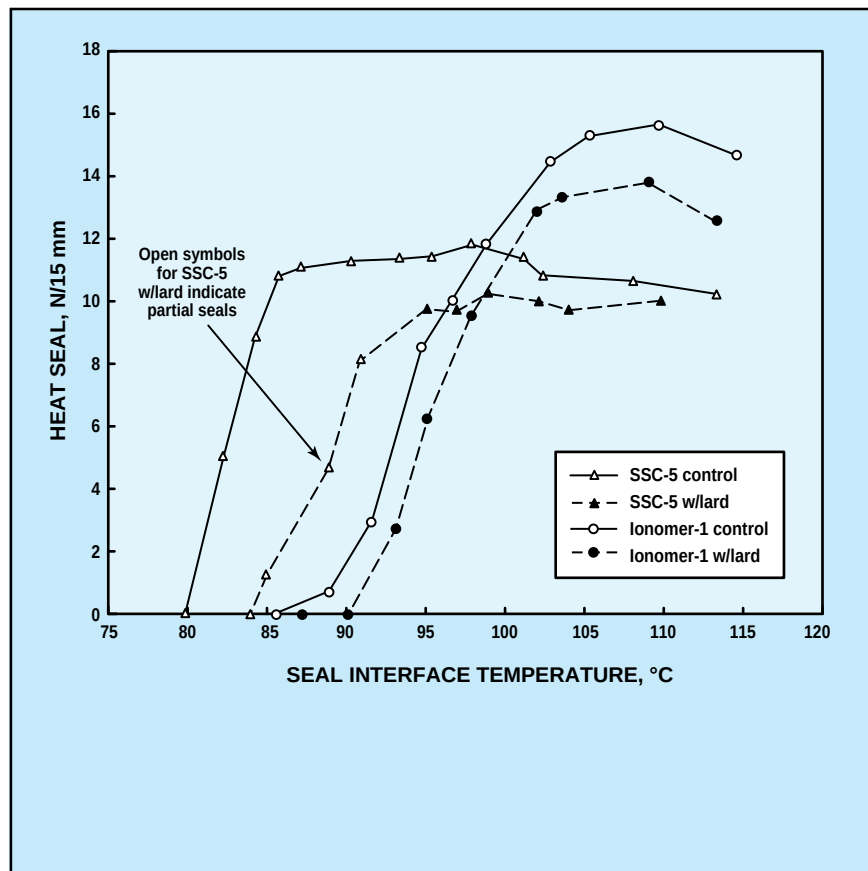
in **Table III** and plotted as a function of melt index in **Fig. 7**. This figure illustrates the significant melt strength advantage of ionomers and acid copolymers over the metallocene resins. These differences are principally related to long-chain branching, hydrogen bonding, and ionic crosslinking. Even the EVA resin has 2–3 times the melt strength of metallocene resins with a similar melt index. Given this perspective, the presence of long-chain branching (12) in the CGCT-type metallocenes provides only a modest improvement in melt strength relative to the SSC types. In fact, the melt strength for LPE-1, the LLDPE/LDPE blend, fell just above the CGCT trend line in Fig. 7.

En route to film property evaluations, each of the polymer samples was fabricated into 50- μ m monolayer films using a 40-mm single-screw extruder having a 16:1 length/diameter ratio and equipped with a 10-cm blown film die having a 0.50-mm die gap. Measurement of extruder conditions such as screw speed, polymer output, and motor amps enabled the calculation of specific output (kg/h/100 rpm) and specific energy consumption (kW·h/metric ton) for each of the resins; the results are presented in Table III. Specific output and extruder energy consumption are presented as functions of melt index in **Figs. 8 and 9**, respectively. The throughput of the CGCT resins appears unaffected by the incorporation of long-chain branching as all the mPE data points fall along the same increasing trend line. All of the high-pressure copolymers fell onto their own trend line that was strongly a function of melt index. The throughput advantage that the ionomer, acid copolymer, and EVA resins maintain over the substantially linear resins becomes even greater with increasing melt index. In terms of energy consumption, the high-pressure copolymers are once again more efficient than the metallocene resins, as illustrated in

12. Comparative heat seal curves



13. Seal-through contamination—SSC-5 and Ionomer-1



14. Seal-through contamination—ACR-1

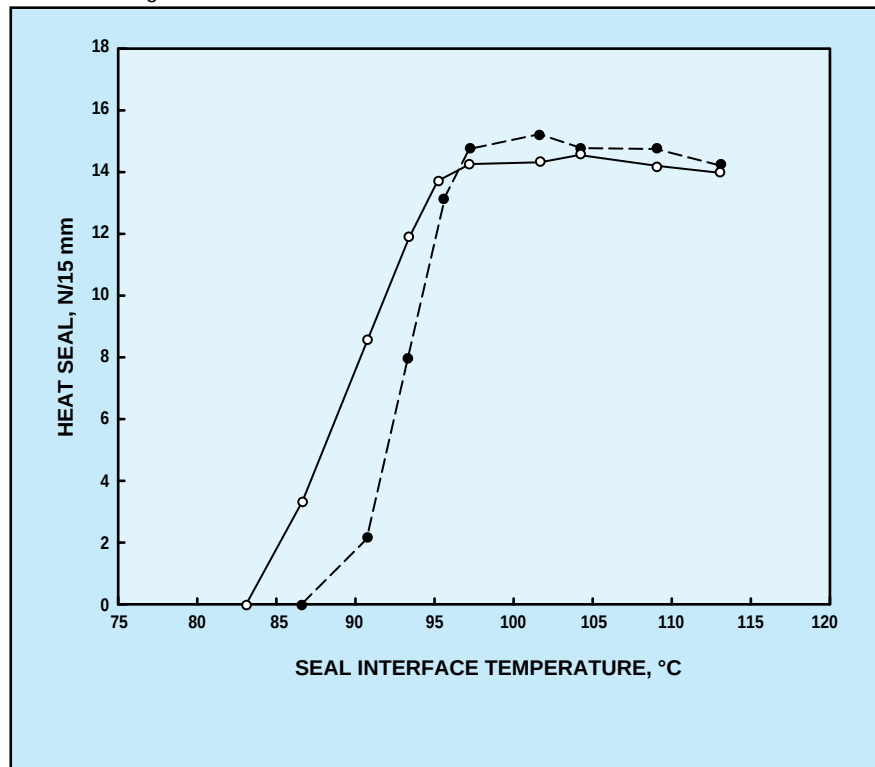


Fig. 9. The SSC resins were the least efficient; however, none of the newly introduced advance-performance terpolymers (APTs) grades (13) were included.

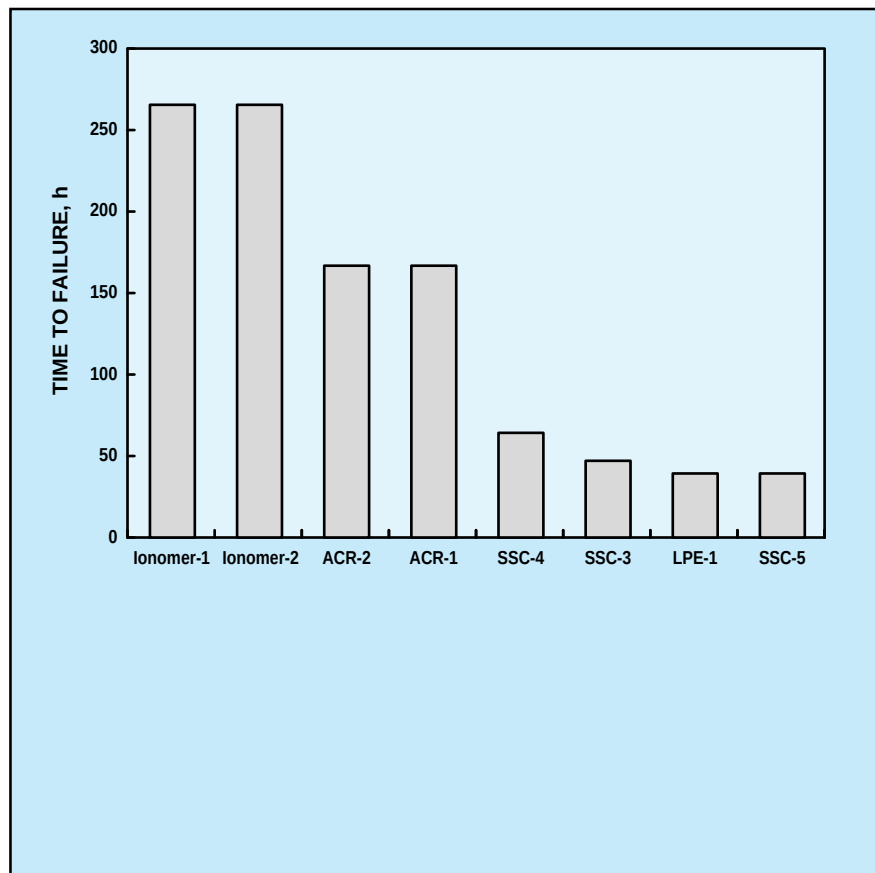
Heat sealability

The heat seal performance of thermoplastic materials is a complex function of time, temperature, and pressure. In a recent paper (14), a model for the sealing of semicrystalline polymers is described to include (a) the time and temperature to allow the opposed polymer surfaces to melt, (b) the pressure to bring these molten surfaces into intimate contact, (c) the time to allow the diffusion and entanglement of opposed polymer chains, and (d) a cooldown period to allow the welded interface time to recrystallize.

Earlier in this paper, it was shown that the compositional homogeneity of the metallocene resins resulted in both sharper endotherms and lower melting points than was previously possible with LLDPE and VLDPE resins (Figs. 5 and 6). Therefore, it was expected that the seal initiation temperatures for the mPE's would also be a stronger function of comonomer composition. The data in **Fig. 10** indicate that the seal initiation temperatures for the metallocene resins can be as much as 15–25°C lower than for the traditional LLDPE and VLDPE resins of comparable compositions. This enhanced sealability now allows for the design of mPE resins that compete as low-temperature sealants with EVA resins in the 12–18% range (**Fig. 11**). Although the low-temperature sealability of ionomers and acid copolymers is challenged by metallocene resins (**Fig. 12**), an 18–33% vinyl acetate EVA would likely be the incumbent material in applications where this criterion alone determines performance.

The value of ionomers and acid copolymers as sealant material goes beyond any single characteristic

15. Corn oil penetration studies (50-μm monofilm at 40°C)



Heat Sealants

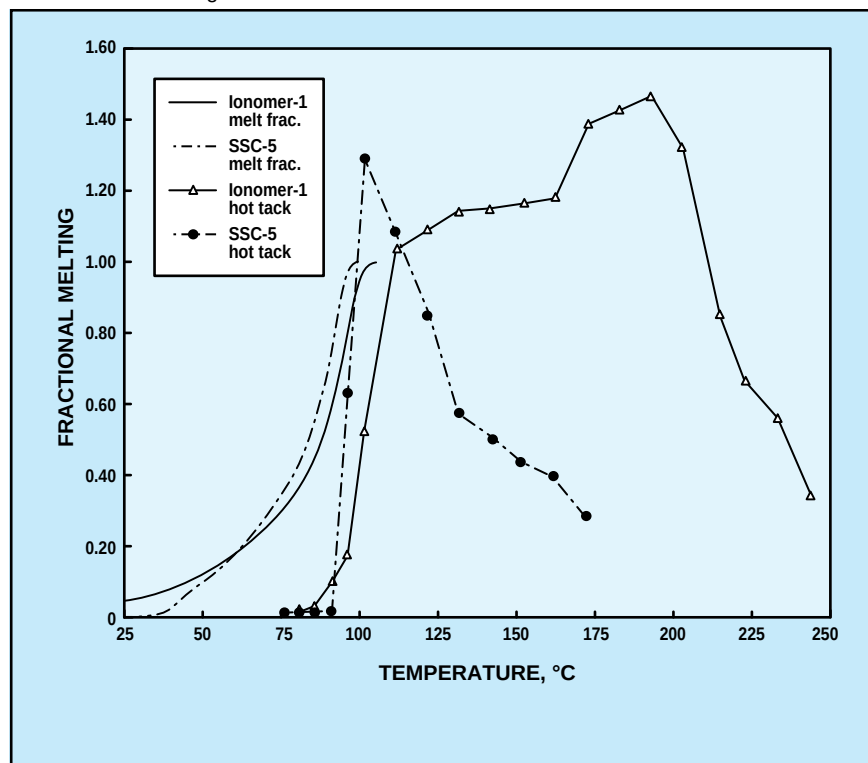
such as seal initiation temperature. The unique chemistry of ionomers and acid copolymers provides for hydrogen bonding and ionic crosslinking, which enhance the polymer chains' ability to diffuse and entangle during the heat seal process. One consequence of these molecular attractions is the ability of ionomers and acid copolymers to seal through fatty contamination. In a demonstration of this characteristic, an evaluation was conducted to judge the seal-through contamination performance of a metallocene resin in comparison to Ionomer-1 and ACR-1. The results, presented in **Figs. 13 and 14**, indicate the negative effect that contamination had on the heat sealability of the metallocene resins. The initial part of SSC-5's heat seal curve was shifted upward 5–10°C in the presence of lard. Furthermore, an examination of the metallocene's seal interfaces upon separation indicated that incomplete seals were being formed. Fatty contamination appears to have prevented the two seal surfaces from coming into full contact with each other, suggesting higher machine failures and in-store leakers with a metallocene sealant.

In contrast, the heat seal curves for Ionomer-1 and ACR-1 were shifted just 2°C and 3–5°C, respectively. The results demonstrate ionomer and acid copolymer effectiveness at sealing through fatty materials. As a result, packages that use high-performance copolymers and ionomers have the highest degree of seal integrity and durability and the lowest rates of machine failures and in-store leakers.

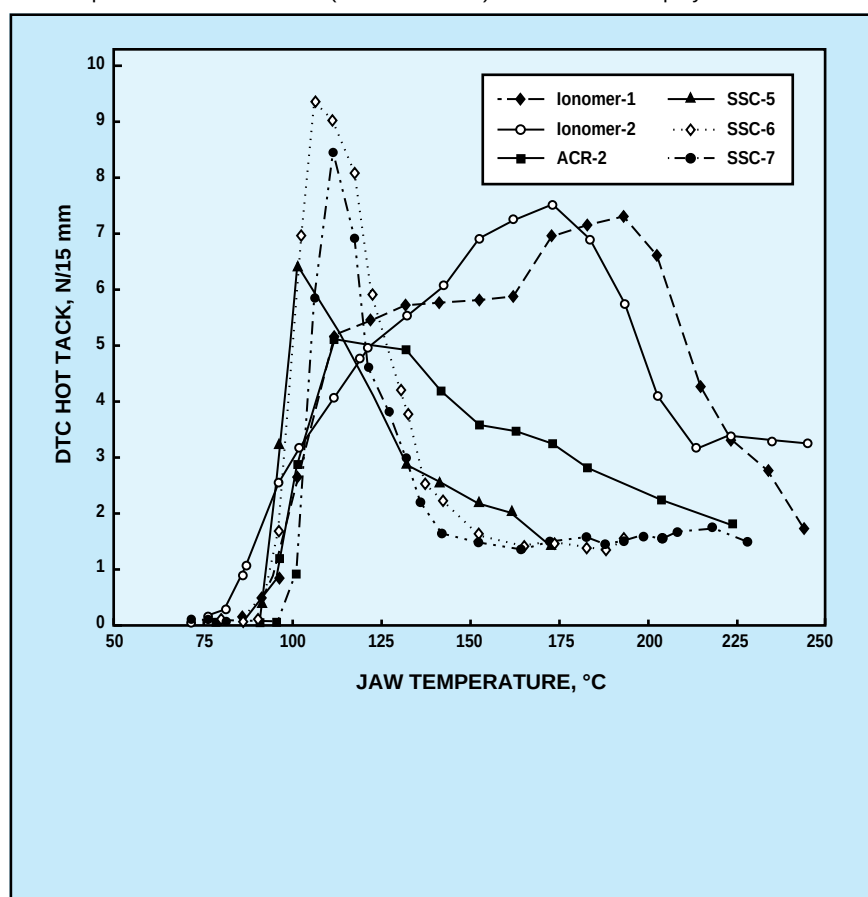
Oil penetration

Another property derived from the chemistry of ionomers is the ability to provide a barrier against grease and oil. The data presented in **Fig. 15** demonstrate that the performance of ionomers, acid copolymers, and polyethylene resins are broken into three distinct levels of perfor-

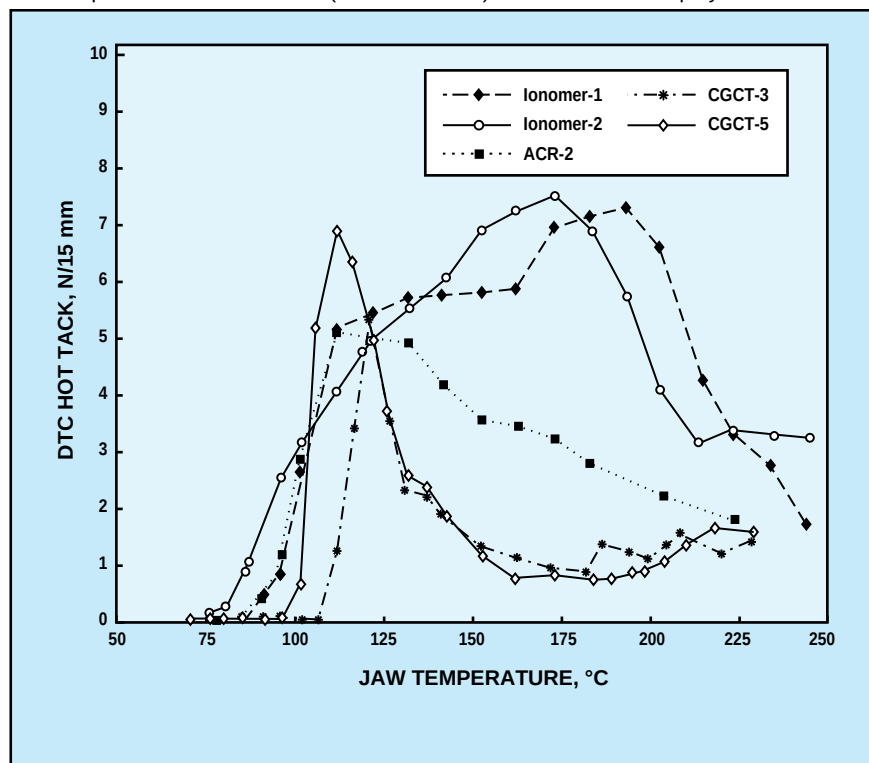
16. Fractional melting and hot tack



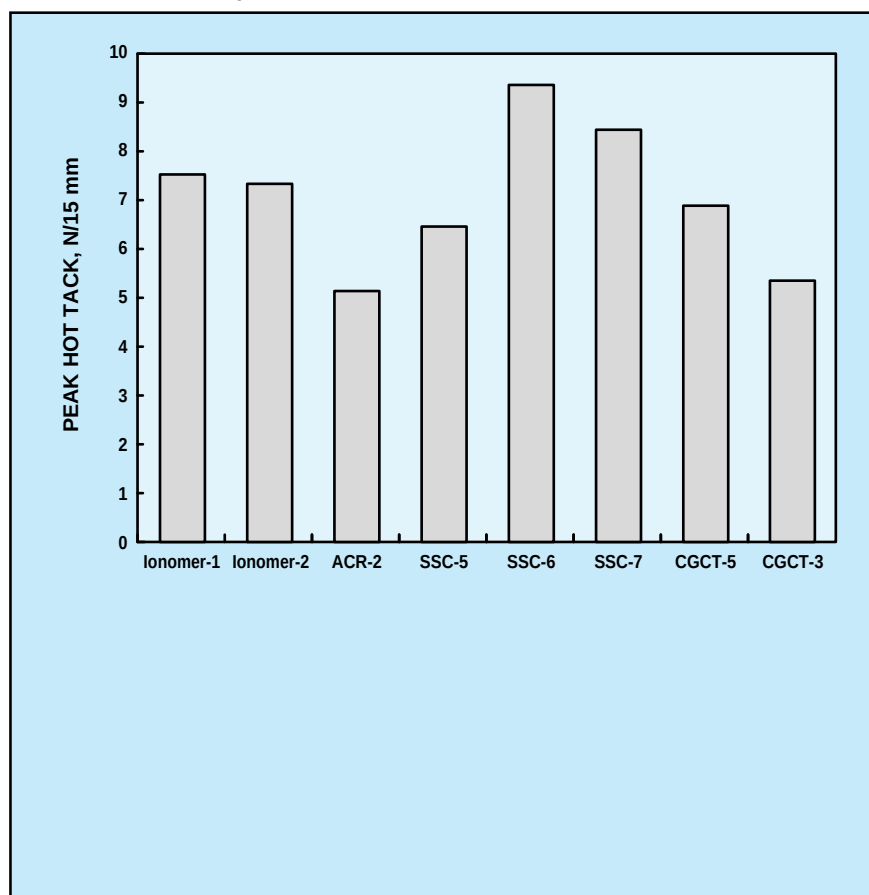
17. Comparative hot-tack curves (DTC Packforsk)—SSC vs. H-P copolymers



18. Comparative hot-tack curves (DTC Packforsk)—CGCT vs. H-P copolymers



19. Peak hot-tack strength



mance. Ionomers are capable of preventing the migration of a typical food oil through a multilayer structure. The acid copolymers, relying upon the acid functionality alone, failed approximately 160 hours into the 264-hour test. Meanwhile, the metallocene polyethylene resins performed no better than the benchmark LPE sample because they are all nonpolar hydrocarbon polymers.

Hot-tack performance

Hot tack is among the most important attributes of any high-performance thermoplastic sealant material. It enables packaging equipment to run at higher line speeds without having to sacrifice seal integrity and package performance. Historically, hot tack has been defined (15, 16) as the ability of the molten seal to resist the forces of separation at the exact moment the seal jaws are opened. The purest form of hot-tack characterization includes those methods that incorporate no delay between the opening of the jaws and the application of force upon the seal.

Unfortunately, rigorous methods such as the spring test require highly trained technicians that must take numerous measurements in the characterization process. In a compromise between test efficiency and the rigorous characterization of hot tack, a Model 52E computer-controlled version of the DTC Hot Tack Tester was used to quantify hot-tack values. Standard test conditions were: 0.5-s seal time, 0.5-N/mm² jaw pressure, and 0.10-s delay time.

The hot tack of ionomers and acid copolymers comes from a mechanism that is quite different than that for linear polyethylene, including the newer metallocene resins. Although all crystallinity is destroyed in the melt phase, the ionic crosslinks and hydrogen bonds of the ionomers and acid copolymers create intramolecular attractions that give these polymers added strength in the melt phase. Previous investigations (17)

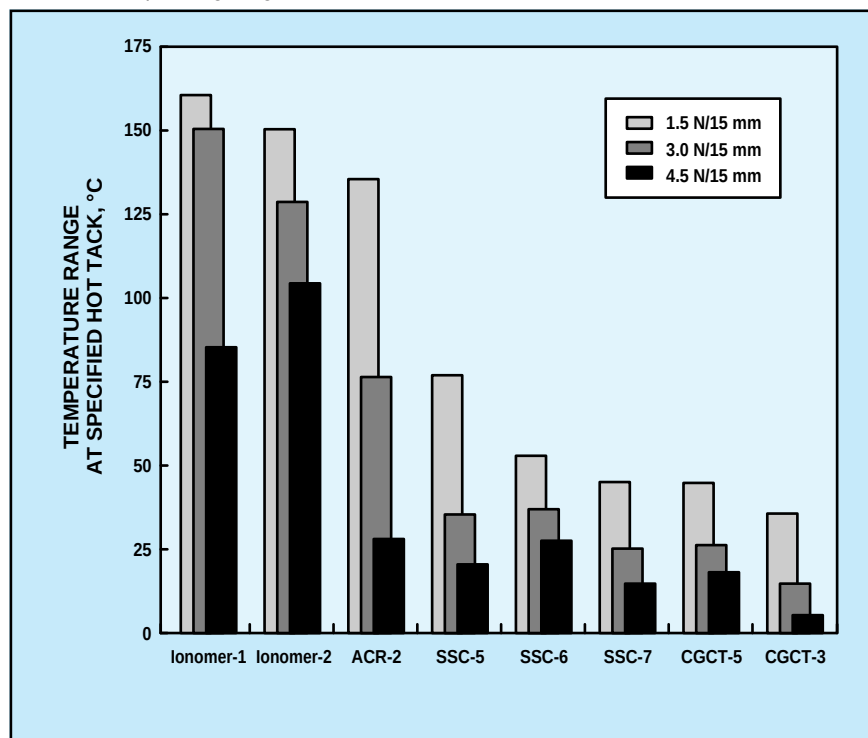
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have shown that the metallocene materials build peak hot-tack strength in a very narrow temperature zone (**Fig. 16**) that begins at about 0.80 fractional melting and ends with complete melting. In addition, the mPE's hot tack drops off rapidly as chain-end mobility (increasing with temperature) and polymer viscosity (decreasing with temperature) shift toward an unfavorable balance. Hot-tack tests that include excessive amounts of delay time result in an increase in hot-tack strength because the polymer interface has time to cool, build viscosity, and then recrystallize.

In contrast, the hot tack of ionomers develops more gradually as energy is required to mobilize the ionic crosslinks; however, once this is accomplished, the rearrangement of ionic clusters across the interface boundary has a great effect on the breadth of the hot-tack curve. As the polymer interface temperature increases, the rearrangement is more effective, with clusters penetrating as much as 200 Å during the short heat seal process (17). This mechanism overcomes the mobility/viscosity balance that must be struck for the mPE's. Although acid copolymers lack ionic crosslinks, they benefit from the hydrogen bonding between the acid groups, and they can provide reasonable performance in less-demanding applications. Once again, the unique polymer chemistry of ionomers and acid copolymers is evident in superior performance.

In **Figs. 17 and 18**, more complete comparisons between the mPE's, acid copolymers, and ionomers are made. Although the metallocene materials can generate high peak hot tack (**Fig. 19**), under these test conditions, they continue to lack the broad hot-tack performance of the high-pressure copolymers (**Fig. 20**). Lab-based hot-tack performance can be used to judge the field performance of a sealant material. Historically, the broad hot-tack operating window for ionomers and acid copolymers has translated

20. Hot-tack operating range



into higher packaging line speeds, reduced seal failures at the machine, and lower in-store leakers.

Conclusions

The unique polymer chemistry of ionomers and acid copolymer resins leads to a balance of attributes such as high melt strength, efficient extrusion processability, versatile heat sealability, oil resistance, and the broadest hot-tack performance that is best suited to both the converter and the packaging end user in premium and high-performance sealant applications.

The results of seal-through contamination evaluations and oil penetration studies confirmed the performance advantage of ionomers and acid copolymers. In hot-tack testing, ionomers demonstrated the broadest level of hot-tack performance opposite a broad selection of metallocene materials that were evaluated. The performance of acid copolymers make them an attractive choice in applications that demand less hot tack.

Many of the enhanced attributes of the metallocene resins are the result of greater compositional homogeneity. In particular, this uniformity results in sharper and lower melting points compared to traditional linear polyethylene materials. As a low-temperature sealant, mPE's could be positioned against incumbent ethylene/vinyl acetate resins.

Finally, the processability of the metallocene materials is judged to be inferior to the high-pressure copolymers. Ionomers, acid copolymers, and EVA resins have substantially greater melt strength, extruder output, and energy efficiencies than the mPE's. The presence of long-chain branching in the "substantially linear" CGCT resins resulted in only limited improvements over the SSC resins. ■

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