



# Engineering Properties and Structure Division Newsletter

April 2017

## CHAIRPERSON'S REPORT



### Dear EPSPDIV Members,

I can't believe ANTEC 2017 is only about one month away! I am excited to see an impressive EPSPDIV technical program that our TPCs Jason Lyons and Brian Grady have outlined. Below is a list of the 8 sessions our division is going to offer at this year's ANTEC:

- M7 Engineering Properties and Structure & Sustainability Join Session (Monday morning)
- M26 From Polymer Processing to Polymer Nanocomposites (Monday afternoon)
- M25 Polymeric Modeling (Monday afternoon)
- T6 Olefin Based Polymers (Tuesday morning)
- T5 Symposium in Honor of Robert A Weiss (Tuesday morning)
- T21 Symposium in Honor of Robert A Weiss (Tuesday afternoon)
- W6 Polymer Characterization and Processing (Wednesday morning)
- W5 Structure and Property of Polymers (Wednesday morning)

### *EPSPDIV to Host 8 Sessions at ANTEC 2017*

In addition to the technical sessions above, we will host a board meeting, a TPC luncheon, and an award reception at ANTEC. You can find the times and locations of these events in the newsletter and we hope to see you there. This year also marks the 75<sup>th</sup> anniversary of SPE. Founded in 1942, SPE is the largest, most well-known plastics professional society in the world. SPE takes action every day to help companies in the plastics industry succeed by spreading knowledge, strengthening skills, and promoting plastics. Employing these vital strategies, SPE has helped the plastics industry thrive for 75 years and counting. SPE has become the recognized medium of communication among scientists, engineers, and technical personnel engaged in the development, conversion, and application of plastics. So let's get together in May and celebrate this big milestone that SPE has achieved.

Besides the upcoming ANTEC, I am also very happy to make a few announcements. First, EPSPDIV just received the Gold Pinnacle Award and the Special Recognition Level Communications Excellence Award from SPE. The Pinnacle Gold is awarded to a division that, in addition to meeting Pinnacle Silver criteria, demonstrates superior performance in the Gold achievement categories. It is a very prestigious award and such a great honor for all of us. Second, our current board member Brian Grady was recently elected as SPE's new President-Elect. Brian has served on our board for a long time and led the membership committee in the past few years. We congratulate Brian and wish him all the best on his

new post. Last but not least, I'm delighted to announce the recipient of last year's best paper award – Yijian Lin from The Dow Chemical Company, for his paper titled HIGH PERFORMANCE HIGH DENSITY POLYETHYLENE (HDPE) FOR HOT FILL CLOSURE APPLICATIONS. We will recognize his work at our reception on Tuesday night.

I look forward to seeing all of you in Anaheim, California very soon!

--- Daniel Liu



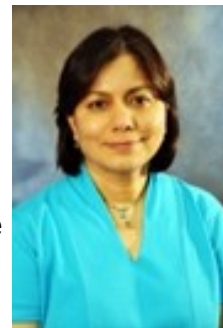
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# COUNCILOR'S REPORT

## Look forward to another 75 years

The 2017 annual technical conference (ANTEC) of the Society of Plastics Engineers (SPE) is almost upon us. In addition to individual and group achievement recognitions, educational, technical and networking activities, a very special event, the 75<sup>th</sup> anniversary of SPE will be celebrated at ANTEC 2017. Make it a point to attend the activity packed ANTEC 2017 to be held May 8<sup>th</sup> to May 10<sup>th</sup> at Anaheim, California.



The Engineering Properties and Structure Division (EPSDIV) will host eight technical sessions, covering a wide range of topics, over the course of three days at ANTEC. **EPSDIV is actively looking for new members to join the division, especially those interested in helping to advance the division over the coming years.** In case you are one of these individuals but are uncertain about how you can get engaged, stop by at any one of the following events to learn more about EPSDIV:

- Board Meeting: Sunday, May 7<sup>th</sup>, 4:00-6:00 PM, Mezzanine 14
- Technical Program Committee Luncheon: Tuesday, May 9<sup>th</sup>, 12:30-1:30 PM, Avila A
- Award Reception: Tuesday, May 9<sup>th</sup>, 5:00-7:00 PM, Avila B

To paraphrase, the purpose of EPSDIV is to educate and promote polymer science & engineering with specific emphasis on

- The investigation and understanding of the engineering properties of polymers and their relationships to composition, structure, and processing variables
- Technical activities on uniform testing and reporting of polymer properties and
- The proper application of property data

These core areas form the foundation of the polymer industry and academia. As such education in, and promotion of these areas should be of universal value to individuals in different stages of their career. Come join the discussion and help make a difference.

See you at ANTEC 2017!

Babli Kapur



# ANTEC 2017 TPC REPORT

In less than a month we will be heading to ANTEC 2017. We have put together an interesting set of 8 sessions on a variety of topical areas that we think you will enjoy.

Travel safe and see you in Anaheim!

Jason Lyons (Arkema)

Brian Grady (Univ. of Oklahoma)

TPC Chairs

## EPSDIV — ANTEC2017 Sessions

- M7 Engineering Properties and Structure & Sustainability Join Session (Monday morning)
- M26 From Polymer Processing to Polymer Nanocomposites (Monday afternoon)
- M25 Polymeric Modeling (Monday afternoon)
- T6 Olefin Based Polymers (Tuesday morning)
- T5 Symposium in Honor of Robert A Weiss (Tuesday morning)
- T21 Symposium in Honor of Robert A Weiss (Tuesday afternoon)
- W6 Polymer Characterization and Processing (Wednesday morning)
- W5 Structure and Property of Polymers (Wednesday morning)

## ***IMPORTANT ANTEC MEETINGS***

### **EPSDIV Board Meeting**

Sunday, May 7th, 4pm to 6pm

Room: Mezzanine 14

### **EPSDIV TPC Luncheon**

Tuesday, May 9th, 12:30pm to 1:30pm

Room: Avila A

### **EPSDIV Award Reception**

Tuesday, May 9th, 5pm to 7pm,

Room: Avila B

# Brian Grady is next President-Elect of the Society

Since 1994, Dr. Grady has been employed by the University of Oklahoma as a faculty member in the School of Chemical, Biological and Materials Engineering and currently is the Conoco-DuPont Professor of Chemical Engineering. He served on the Executive Committee of SPE from 2008-2012 and served as Secretary of the Society in 2010-2011. He was elected as a Fellow of the Society in 2012. He received a B.S. from the University of Illinois in 1987, and a PhD from the University of Wisconsin in 1994, both in Chemical Engineering. Nominated by the Engineering Properties and Structures Division.



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***Congratulations Brian!***

***The Engineering  
Properties and  
Structure Division  
appreciates your  
many years of  
volunteer service!***

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# TREASURER'S REPORT

## FINANCIAL REPORT FROM JULY 1, 2015 TO JULY 15, 2016

**BALANCE as of July 1, 2015** **36,195.96**

(cash, checking, savings, investments)

<b>INCOME</b>	<b>ACTUAL</b>
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SPE Rebate	2230.14
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ANTEC Sponsorships	1466.10
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<b>TOTAL INCOME</b>	<b>\$3,696.24</b>
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### EXPENSES

Student Travel Fund	2,500.00
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International Award	2,500.00
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ANTEC Expenses	275.00
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ANTEC TPC	4,171.19
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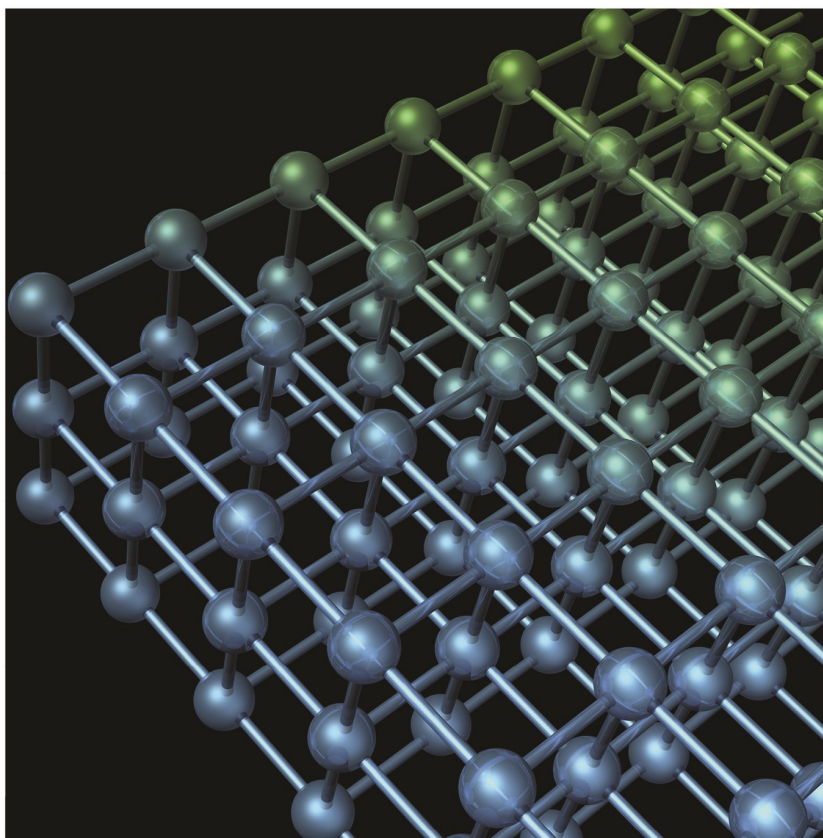
Bank Fees	33.00
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<b>TOTAL EXPENSES</b>	<b>\$9,479.19</b>
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**ENDING BALANCE as of July 15, 2016** **30,413.01**

*Submitted by Emmett Crawford,  
EPSDIV Treasurer 2015-2016*



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# 2017 EPSDIV ANTEC Technical Program Sponsorships

## Sponsorships Still Available for the 2017 ANTEC Program!

**Sponsorship  
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- ◆ Your Company logo on screen between talks!
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**...as of April 12th**

Funds from the program are used to support the EPSDIV mission in two key areas:

#### ◆ **Technical conference program quality**

Such as, sponsorship of awards for outstanding papers, expenses and honoraria for invited speakers, and seed money for RETECs and other EPSDIV sponsored symposia.

#### ◆ **Plastics education**

Such as, sponsorship of awards for outstanding student papers, student travel grants, scholarships and short courses.

**Please ask your company or organization to sponsor the  
2017 ANTEC Technical Program—Thanks!**



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# Congratulations to Yijian Lin from the Dow Chemical Company, selected as ANTEC 2016 Best Paper

## HIGH PERFORMANCE HIGH DENSITY POLYETHYLENE (HDPE) FOR HOT FILL CLOSURE APPLICATIONS

*Yijian Lin, Babli Kapur, John Sugden  
The Dow Chemical Company*

### Abstract

High performance bimodal high density polyethylene (HDPE) was developed for the hot fill closure applications. Performance of the bimodal HDPE was benchmarked versus incumbent unimodal HDPE resins. The bimodal HDPE resin delivered better environmental stress cracking resistance (ESCR) than a conventional HDPE homopolymer while maintaining a good heat deflection temperature (HDT) and a good Vicat softening point. The high performance HDPE also exhibited greater shear thinning behavior, indicative of good processability under the high shear rates typically encountered in the injection molding process. In addition, the closures made from the new HDPE resin are advantaged with respect to the removal torque.

### Introduction

Traditionally beverage closures are made from polyolefins consisted of 2 pieces, with a shell fabricated from polypropylene (PP) and a liner made from elastomers. The ongoing trend towards conversion from 2-piece PP closures to 1-piece liner-less high density polyethylene (HDPE) closures has a significant value in terms of raw material cost saving and supply chain efficiencies [1].

Sustainability and increasing consumer health consciousness have had a significant impact on how beverages are produced and packaged today. The sustainable packaging has resulted in reduction of material used to produce the package, and furthermore, highlighted the importance of recycling. The increased health consciousness has shifted consumer preference towards the healthy beverages such as milk, iced tea, fruit juices, energy drinks, and sports drinks that are produced without preservatives.

In order to maximize shelf life, these beverages as well as their packages are subjected to various sterilization processes such as aseptic, hot fill, and pasteurization. In the hot fill processes, the beverage is typically heated up to 95°C for 20 to 30 seconds and then filled into bottles at a temperature of around 85°C. After applying the closures, the package is inverted for about 15 to 25 seconds, allowing the closures to contact with the 85°C liquid for a time period of sterilization. It is critical

for the closures to maintain their rigidity and functionality at elevated temperatures to keep the contents sealed under the heat and pressure throughout the entire process.

In this study, a high performance bimodal HDPE resin was developed using The Dow Chemical Company proprietary technology. Performance of the new resin was benchmarked versus the incumbent unimodal HDPE resins. The environmental stress cracking resistance (ESCR), heat distortion temperature (HDT), Vicat softening point, removal torque, as well as the processability of the bimodal HDPE resin were investigated.

### Materials and Experimental

Three HDPE resins were used in this study. Melt index (MI) and density of the resins are listed in Table 1. HDPE-1 was an ethylene homopolymer while HDPE-2 was an ethylene-hexene copolymer. HDPE-3 was a bimodal copolymer resin containing two different polyethylene components that were blended inside the reactor at molecular level.

**Table 1. Properties of the HDPE resins**

Resin	Modality	I <sub>2</sub> (g/10min)	Density (g/cc)
HDPE-1	Unimodal	8.2	0.965
HDPE-2	Unimodal	10	0.952
HDPE-3	Bimodal	3.2	0.964

MI was measured according to ASTM Method D1238 at 190 °C and 2.16 kg. Density was determined by liquid displacement (Archimedes Principle) using 2-propanol as solvent in accordance with ASTM D792 Method B.

Melting behavior of the samples was obtained from differential scanning calorimetry (DSC). A TA Instruments Q1000, equipped with a refrigerated cooling system and an autosampler was used to perform this analysis. During testing, a nitrogen purge gas at a flow rate of 50 ml/min was used. First, the sample was rapidly heated to 180°C, and held isothermal for five minutes, in order to remove its thermal history. Next, the sample was cooled to -40°C, at a 10°C/minute cooling rate, and held isothermal at -40°C for five minutes. Then, the sample was heated to 150°C at a 10°C/minute heating rate.

Crystallinity ( $X_c$ ) of the sample was calculated according to

$$X_c = \Delta H_f / \Delta H_0 \quad (1)$$

where  $\Delta H_f$  is the heat of fusion of the resin obtained from the DSC 2<sup>nd</sup> heating curve.  $\Delta H_0$  is the heat of fusion of 100% polyethylene crystal with a value of 290 J/g [2].

HDT was tested according to ASTM D648(A) at 0.455MPa. Vicat softening point was measured according to ASTM D1525 at a load of 10 N and a heating rate of 2°C/min.

Closures were fabricated via injection molding process. ESCR of the closures was measured as illustrated in Figure 1. The closures were applied on polyethylene terephthalate (PET) bottle finishes at a torque of 18 in.-lbs. The bottle finish part of the assembly was outfitted with tubing to pressurize the air inside the tube to 6 bars. The closures were fully immersed in a 10% Igepal CO630 solution. The time it took for a crack to develop was recorded. Testing temperature was set at 40°C.

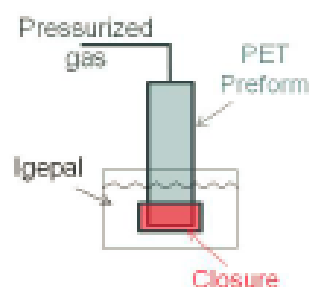


Figure 1. Schematic ESCR testing setup

Rheological properties were measured using a TA Instruments ARES rheometer. Frequency sweeps were run from 0.1 to 100 rad/s in plate-plate mode at 190°C in a nitrogen atmosphere. Diameter of the plates was 25 mm.

Capillary viscosity was measured at 190°C on a Rosand RH 2000 fitted with a flat entrance (180 degrees) die with a length of 16 mm and a diameter of 1 mm at apparent shear rates ranging from  $10^2$  to  $2 \times 10^3 \text{ s}^{-1}$ . Rabinowitsch correction was applied to account for the shear thinning effect. The corrected shear rate and shear viscosity were reported.

Removal torque of the injection molded closures was measured with a Steinfurth AFS 500 torque tester. All closures were applied on the bottles to the same rotation angle and ensure a proper seal before the removal torque measurement took place.

## Results and Discussion

In order to withstand the hot fill processes, the closures need to have an excellent high temperature resistance. Rigidity of the closures needs to be sufficient at elevated temperatures to maintain its seal functionality. DSC was employed to investigate the thermal behavior of the HDPE resins. The 2<sup>nd</sup> heating curves of the resins are shown in Figure 2. HDPE-1 (homopolymer) showed a melting temperature peak at 133.5°C and a crystallinity of 82%. For HDPE-2 (copolymer), the comonomer interrupted the crystallization process of ethylene units and resulted in smaller and imperfect crystals. Consequently, a lower melting temperature at 129.7°C and a lower crystallinity of 72% were observed for HDPE-2. The bimodal HDPE-3 also exhibited a higher melting peak temperature at 132.9°C and a higher crystallinity of 78% than HDPE-2. The melting temperature and degree of crystallinity of these samples agreed well with their measured density.

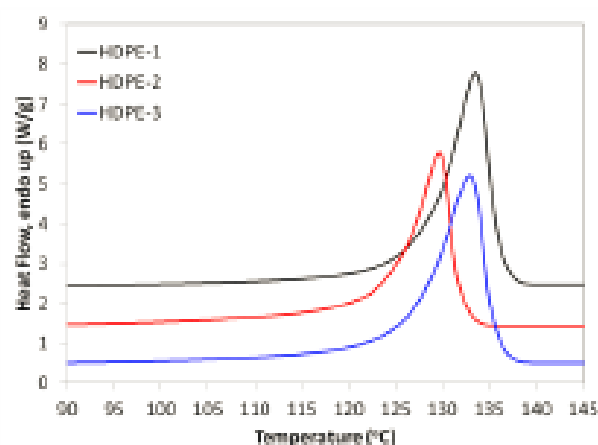
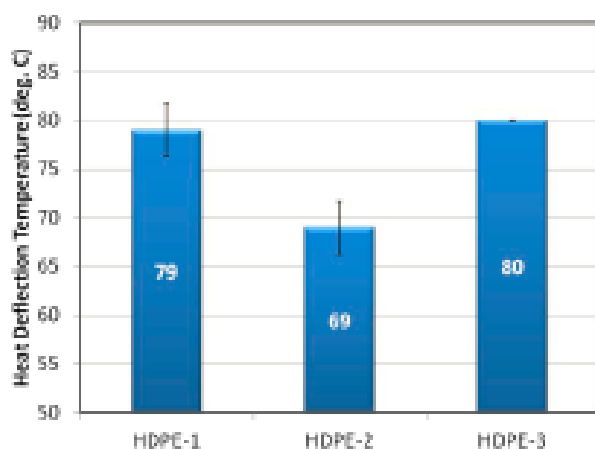
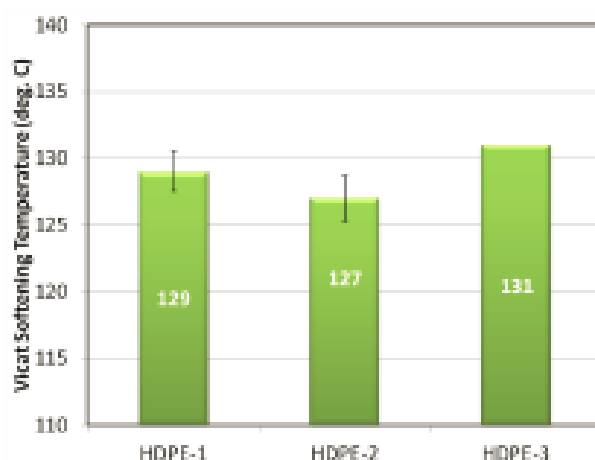


Figure 2. DSC thermograms of the HDPE resins (2<sup>nd</sup> heating curve)

Figure 3 and Figure 4 show the HDT and Vicat softening point of the resins, respectively. The HDT and Vicat softening point reflect the rigidity of a material at elevated temperatures. HDPE-2 showed both the lowest HDT and the lowest Vicat softening point due to its lower crystallinity and imperfect crystals as indicated by the DSC results. HDPE-1 homopolymer showed a higher HDT and a higher Vicat softening point than HDPE-2. For polyethylene resins, the highest crystallinity one can achieve is with a homopolymer. For the best high temperature performance, homopolymer is often used to fabricate the articles. Compared to HDPE-1 homopolymer, HDPE-3 had shown a similar HDT and a slightly higher Vicat softening point. This indicates that the thermal resistance of HDPE-3 is quite similar to the HDPE-1 homopolymer.



**Figure 3. Heat deflection temperature of the HDPE resins.**

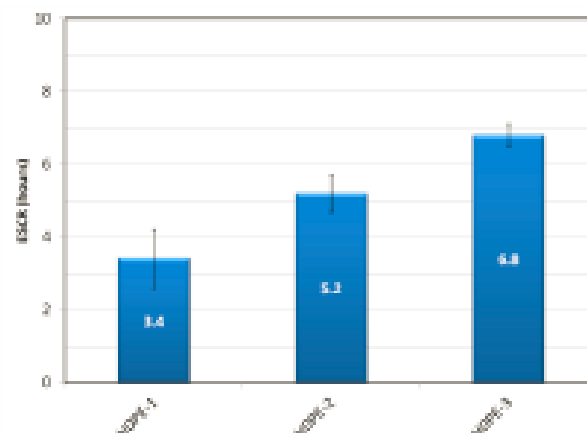


**Figure 4. Vicat softening point of the HDPE resins**

Although a homopolymer has the best high temperature properties, it is usually very brittle due to its lack of amorphous tie molecules that bridge crystals. In closure applications, a good ESCR property is also desired. A good ESCR property can prevent the crack cause by over-tighten and ensure a long term reliable performance. It also allows the closure manufacturers to light weight the closures for cost saving while maintaining the same closure performance.

Figure 5 shows the ESCR performance of the closures made from the HDPE resins. Not surprisingly, the HDPE-1 homopolymer showed the lowest ESCR at an average failure time of 3.4 hours. Although the high crystallinity of the homopolymer enhanced its high temperature performance and stiffness, it also caused the sample to be too brittle and hence have a poor ESCR. In contrast, the HDPE-2 copolymer showed a better ESCR with an average failure time of 5.2 hours. The greater amount of amorphous phase fraction of HDPE-2 provided more tie molecules that improved the toughness of this resin.

Among all three samples, the bimodal HDPE-3 again exhibited the best ESCR property with an average failure time of 6.8 hours. One of the advantages of a bimodal resin is that it can be designed in such a way that one component provides the high temperature property and the other component provides the ESCR property. By mixing the two components at molecular level inside the reactor, both properties can be achieved simultaneously.



**Figure 5. ESCR performance of the closures**

Another critical property for closure resins is the shear viscosity. A low shear viscosity is required for easy process.

Figure 6 shows the shear viscosity of the resins in the shear rate range of 0.1 rad/s to 100 rad/s. As expected, the viscosity ranked in the reverse order of their MI, with a higher MI being a lower viscosity. All three samples showed non-Newtonian fluid behaviors, i.e. the shear viscosity decreased as the shear rate increased. The rate dependant viscosity of polymers corresponds to the disentanglement of molecular chains over a given experimental time scale. In theory, the onset of shear thinning is determined by the time required for the longest chain to relax in the melt, and shifts to a lower frequency with increasing molecular weight distribution (MWD). The degree of shear thinning, which can be defined as the ratio of the viscosity at 0.1 rad/s to the viscosity at 100 rad/s, increases with the MWD also [3].

HDPE-3 exhibited a higher degree of shear thinning compared to the two unimodal HDPE resins (see Figure 6) as a result of the bimodal molecular weight distribution. The onset of shear thinning of HDPE-3 also started at a lower frequency than HDPE-1 and HDPE-2 due to the longer relaxation time of the high molecular weight component in the bimodal design. Average relaxation time of the three samples was also calculated by fitting the rheology curves using the Cross model [4]

$$\eta^* = \eta_0 / (1 + (\lambda \cdot \omega)^{1-n}) \quad (2)$$

where  $\eta^*$  is the complex shear viscosity,  $\eta_0$  is the zero shear viscosity,  $\lambda$  is the average relaxation time,  $\omega$  is the shear rate, and  $n$  is the relaxation exponent.

Table 2 summarizes the rheological parameters of three HDPE resins. HDPE-3 showed the longest average relaxation time due to its high molecular weight component in the bimodal design. The zero shear viscosity provides a further tool to probe molecular structure because it is insensitive to the MWD and should most significantly depend on the weight average molecular weight [5]. Clearly, the HDPE-3 had a much higher zero shear viscosity than the HDPE-1 and HDPE-2 which was consistent with its lower melt index and higher weight average molecular weight.

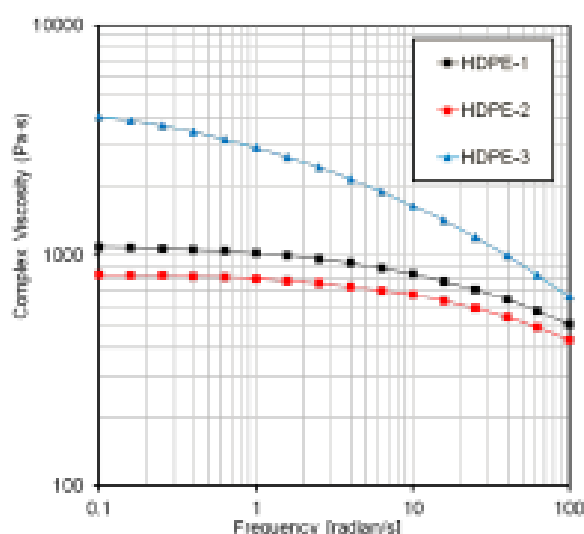


Figure 6. Complex viscosity of the HDPE resins as a function of shear rate

Table 2. Rheological parameters of the HDPE resins

	$\eta_0$ (Pa-s)	$\lambda$ (s)	$n$
HDPE-1	1120.5	0.01456	0.4435
HDPE-2	845.4	0.00938	0.4184
HDPE-3	4722.4	0.35277	0.4981

During the fast speed injection molding process, the polymer melt is subjected to a very high shear rate. Therefore, a stronger shear thinning rheological behavior is always desired.

The capillary rheometer provides further information into the higher shear rate range which is more relevant to the injection molding process. Figure 7 shows the shear viscosity of the HDPE resins in the shear rate range of  $10^2$   $s^{-1}$  to  $2 \times 10^5$   $s^{-1}$ . Although the HDPE-3 had a lower MI and a higher shear viscosity in the low shear rate range, due to its strong shear thinning effect, the viscosity of HDPE-3 was similar or even slightly lower than the

HDPE-1 and HDPE-2 in the shear rate range of greater than  $500$   $s^{-1}$ . This indicates that the HDPE-3 can be processed similarly to the HDPE-1 and HDPE-2 during the injection molding process.

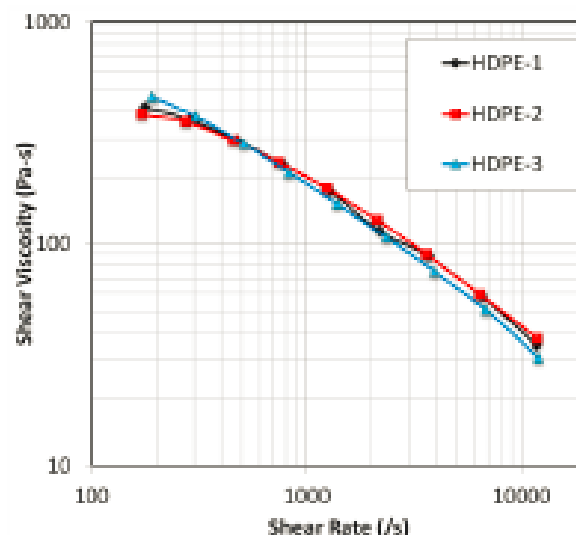


Figure 7. Capillary shear viscosity of the HDPE resins

Figure 8 shows the removal torque of the closures after they were applied on the bottles. Removal torque is the maximum torque to initially rotate the closure for release. For the sake of easy open, a lower removal torque is often desired. HDPE-1 and HDPE-3 showed lower removal torque than the HDPE-2.

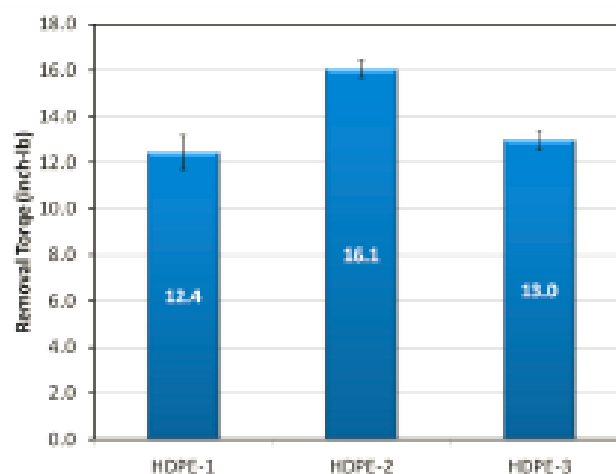


Figure 8. Removal torque of the closures

## Conclusions

Three HDPE resins were studied in terms of the performance for the hot fill closure applications. It was found that the HDPE homopolymer, due to its higher crystallinity, showed better high temperature properties such as a higher HDT and a higher Vicat softening point when compared to the HDPE copolymer. However, the



ESCR performance of the HDPE homopolymer was compromised. The bimodal HDPE resin developed for the hot fill closure applications delivered better environmental stress cracking resistance (ESCR) than the HDPE homopolymer while maintaining the HDT and Vicat softening point. The bimodal HDPE also exhibited greater shear thinning behavior, indicative of good processability under the high shear rates typically encountered in the injection molding process. In addition, the closures made from the new HDPE resin are advantaged with respect to the removal torque.

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