SLIP LEVEL AND APPLICATION ANGLE VERSUS TORQUE-CONSTANT CLOSURE APPLICATION

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Abstract

The purpose of this paper is to analyze the effects of material choice on torque removal by comparison of torque-constant and angle-constant application methods and subsequent removal torques over time. The material parameter varied for the purpose of this study was the slip level of a bimodal high-density polyethylene.

Background

The traditional rule of thumb for determining application torque is simple:

$$application\ torque\ (in.lbs.) = \frac{cap\ diameter\ (mm)}{2}$$

The cap diameter in millimeters divided by two gives an approximate application torque in inch.pounds. While this estimate works well for many caps and bottles, as the number of closure designs, materials and combinations thereof increase, robust torque application studies are advisable to determine the optimal closure application method. This includes an application torque value and, or angle value for specific package designs.

Without a comprehensive study, the opening torque, seal integrity and as a result, the final product quality and customer experience may be adversely affected.

To determine the appropriate application torque or application angle, a preliminary step is to review the most current application torque reference tables [1] to get an idea for widely accepted ranges of application values. It is important to reference one that includes information about the cap and bottle material composition, the seal design, pressure retention, and other important parameters.

Next it is important to work closely with the cap or resin supplier to understand the unique qualities of the material used to create the closure. Factors such as modality, colorant type and level, and additive composition (presence or absence of slip, nucleator, mold release agent, etc.) can greatly impact not only how the material processes, but also the final part physical properties and how it performs over its service life.

Once the packaging materials are understood, testing equipment (automatic torque analyzer, submersion leak tester) can be used to determine the following process variables for each package design:

<u>Strip torque (Tstrip)</u>
The torque value at which the closure and, or bottle threads fail during capping due to over-torqueing. It is very important for production line technicians to be aware of the strip torque value and understand the consequences of over-torqueing (wrinkles on induction seal, damaged threads, loose cap, etc.).

<u>Maximum Seal Torque ($Tseal_{max}$)</u>
The maximum torque that may be applied without causing wrinkles on induction seals.

<u>Minimum Seal Torque ($Tseal_{min}$)</u>
For induction sealed closures, this is the minimum torque that must be applied in order to provide sufficient contact between the opening of the container and the induction seal. For non-induction sealed closures, this is the torque at which the 1-piece or 2-piece closure seals the bottle. The seal integrity should be tested using bubble emission or dye leak detection methods. It is important to consider the stresses encountered during product transportation, such as vibration and internal pressure changes, as well as the dwell time (shelf life, accelerated aging) of the product when evaluating seal integrity and setting $Tseal_{min}$ values/limits

<u>User torque limits ($Tuser_{min}$ and $Tuser_{max}$)</u>
To evaluate the consumer group's closure opening experience, caps must be tightened to different set-points, and the target group must rate the samples in each batch for ease of opening. This evaluation will result in $Tuser_{min}$ and $Tuser_{max}$ values. The range between $Tuser_{min}$ and $Tuser_{max}$ can be referred to as the consumer group's "opening torque comfort zone".

<u>Torque Upper Spec Limit (Tusl)</u>
The upper spec limit for application torque.

<u>Torque Lower Spec Limit (Tlsl)</u>
The lower spec limit for application torque.

To determine Tstrip, $Tseal_{max}$, $Tseal_{min}$, $Tuser_{max}$ and $Tuser_{min}$, batches of closures should be tightened to 25%, 50%, 75%, 100%, 125%, 150%, 175% of the application set-point recommended by the cap supplier and evaluated as described above for each variable.

For a well-controlled capping process, the following equations should be satisfied:

$$Tset\_point = Tseal_{min} + \frac{Tseal_{max} - Tseal_{min}}{2}$$

Where

$$Tusl \leq Tseal_{max}$$

and

$$Tlsl \geq Tseal_{min}$$

such that

$$Tseal_{min} < Tset\_point \leq Tseal_{max} < Tstrip$$

As described in the above relationship, it is important to ensure that the torque set point (Tset_point) falls between the minimum and maximum user torque limits and minimum and maximum seal torques so that the bottle seal is ensured and the closure remains comfortable to remove for the user.

By using a torque analyzer equipped with a rotary encoder, the angular position of the cap for each of the above torque parameters can be determined. Understanding not only the torque but also the angle specifications is important because application angle can be used for the verification of vision system based seal integrity testers.

As opposed to using torque removal measurement as part of quality assurance, visual application angle measurement allows testing 100% of the production in a non-destructive manner.

An increasing number of closures and bottles are now marked with special molded-in features to indicate the closure angle. These marks are intended to support both the historical manual application angle measurement and the new non-destructive vision system based approach for the purpose of visual seal integrity testing (Fig. 1).

The mark on the outside of the cap indicates the thread start of the closure whereas the mark on the bottle neck indicates the thread start on the bottle. The distance between the two marks gives an indication of how far the closure has been tightened onto the neck finish threads (Fig. 2, 3, 4).

Figure 1. Example Setup: Visual Application Angle Measurement

Figure 2. Application Angle Suggests Torque is too High

Figure 3. Application Angle Suggests Torque is Close to Target

Figure 4. Angle Suggests Torque is too Low

While visual application angle inspection systems can test 100% of the packages non-destructively, it is important to note that due to variation in the plastic composition, bottle and cap molding processes, and possible mechanical damage to the seal area, the external appearance of the marks may not always indicate seal quality accurately. For this reason, it is suggested that visual application angle testing be complemented by regular leak and torque testing.

Analysis of Non-Destructive Release Angle Range of a 1-Piece Closure

For the purposes of this paper, non-destructive release refers to measuring thread break torque without breaking the seal. Depending on seal type (1-piece vs. 2-piece closures), application torque, angle and internal pressurization, the non-destructive release angle will vary.

During the first trial, bottle "A" was pressurized to 5 psi and the closure tightened with 10 in.lbs torque. Upon submersion leak testing of the seal, the package was determined to have insufficient seal (Fig. 5).

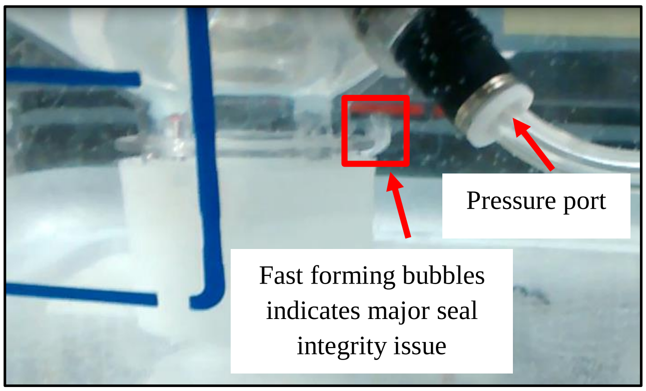

Figure 5. Submersion Leak Testing of Closure Applied to Bottle "A" with Torque of 10 in.lbs Pressurized to 5 psi.

The torque application procedure was replicated on Bottle "A" with the same closure using 20, 30 and 40 in.lbs, and maintaining the 5 psi internal pressurization. Varying decreasing leak rates were recorded and finally at 50 in.lbs, Bottle "A" was sealed.

Following this procedure, non-destructive thread break torque testing was performed. It was determined that between 15 to 30 degrees of removal angle compromised the seal. After further trials, the non-destructive removal torque testing was performed at 8 degrees to avoid compromising the seal.

An alternate closure was applied to the same bottle and similar performance to what was described above was observed.

Following this, 4 more containers "B, C, D, E" were tested by the same methods as above. Bottles "B" through "E" maintained seal as low as 10 in-lbs of application torque. At 10 in.lbs application torque, the closures maintained the seal up to 15 degrees of counter-clockwise (removal) rotation. At 50 in.lbs application torque, the closures maintained seal up to 45 degrees of counter-clockwise (removal) rotation. The results of Bottle "A" did not match the results achieved by submersion leak testing Bottles "B" through "E".

To investigate the different submersion leak testing behaviors of Bottle "A" versus Bottles "B" through "E", the bottle openings were visually inspected. It was discovered that Bottle "A" had a small surface inconsistency, or chip, at the inside of the lip (Fig. 6), and cracking at the outside of the lip (Fig. 7). These defects likely resulted from rough handling (impact to bottle during shipping) prior to capping.

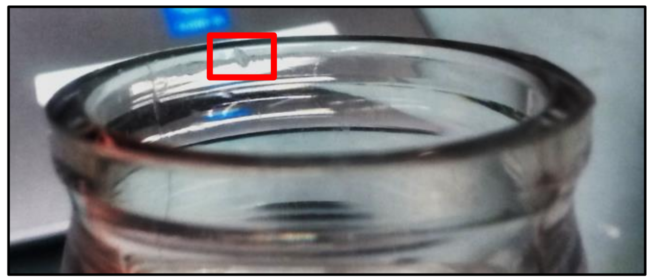

Figure 6. Small Surface Defect on Inside of Container Opening

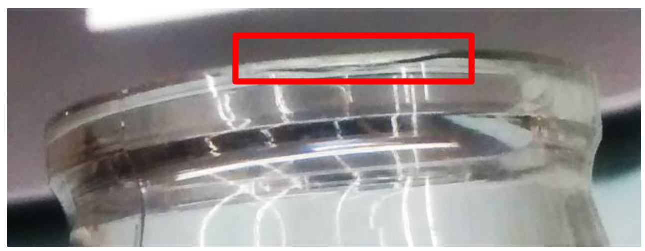

Figure 7. Cracking at the External Edge

The leak testing performance in this study is indicative of one issue with visual application angle measurement as means of seal integrity verification. Minor mechanical defects to the neck are not observable externally and such defects while not affecting the application angle can easily compromise the seal integrity while going unnoticed by visual inspection systems.

If for a given period of time a significant percentage of bottle neck finishes were compromised by surface deformity, visual application angle inspection can potentially overlook widespread seal integrity issues unless the Quality Assurance procedure included regular leak testing.

Experiment

Analysis of the Effect of Varying Levels of Slip Agent on Application and Removal Torque Performance

Materials

Three samples of a natural bimodal high-density polyethylene (HDPE) resin were used in this study. The samples are identified throughout the paper by the following:

No Slip
Resin prepared without a slip agent present.

Low Slip
Resin prepared with 50% of the amount of slip used in the Standard Slip sample.

Standard Slip (Std. Slip)
Resin prepared with what is considered by the industry to be a standard level of slip agent for HDPE closures designed for carbonated beverages.

Each sample was made using bimodal HDPE copolymer flake with identical melt index, density, catalyst, and reactor production conditions to ensure consistent performance between samples. The only factor varied between each sample was the additive package to produce no slip, low and standard slip samples. The slip agent used in the low and standard slip samples is the same mono-unsaturated primary amide.

Bimodal HDPE was chosen as base material for this study due to the increasing importance of bimodal materials in light-weighted closures. Bimodal HDPE provides a significant improvement in environmental stress cracking (ESCR) (Fig. 8) over similar monomodal resins while retaining the necessary stiffness to resist deformation by pressurized contents. Therefore, greater closure light-weighting can be achieved with bimodal HDPE versus monomodal HDPE.

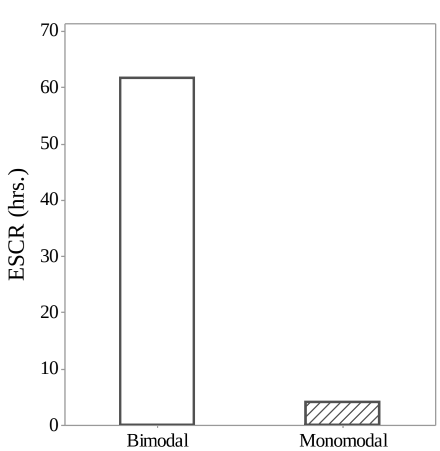

Figure 8. Monomodal vs. Bimodal Comparison: ESCR (ASTM D1693 [2] Condition B, 100% Igepal, F50) at Comparable Stiffness

Polyethylene terephthalate (PET) preforms were used to simulate bottle neck finishes for the purpose of this torque removal study.

Procedure

Closure Molding Conditions

Table 1. Injection-Molded 28 mm Cap

Screw and Nozzle Temperature	220°C
Hot Runner Temperature	230°C
Cycle Time	9.60 seconds
Shot Weight	6 x 2.90 grams
Mold Fluid Temperature	10°C

The closures were conditioned at room temperature for approximately 1 week following production.

Preform Molding Conditions

Table 2. PET Preform Molded by SGT France

Screw and Nozzle Temperature	280°C
Hot Runner Temperature	280°C
Cycle Time	12.0 seconds
Shot Weight	72 x 35 grams
Mold Fluid Temperature	15°C

Torque Testing Equipment

A SureTorque ST-120S automated torque tester was used in this study to evaluate application and removal torque and angle measurements. An automated torque tester was chosen over a manual torque tester to control for a number of process variables, including top-load, bottle and cap gripping pressures, angular speed and torque ramp [3].

Prior to conducting the study, the ST-120S (Fig. 9) was calibrated using NIST traceable dead weights and pulleys, according to ASTM D3474, *Practice for Calibration and Use of Torque Meters Used in Packaging Applications* [4]. The built-in digital pressure transducers were verified using a calibrated pressure gauge, and the accuracy of the 20000CPR rotation sensor was verified using a protractor.

Figure 9. The ST-120S Automated Torque Tester

The process variables, cap gripping pressure, bottle clamping pressure and downward pressure on the closure were controlled as described below.

<u>Cap Gripping Pressure:</u>
To avoid the cap slipping at ~20 in.lbs of application torque and overcome torque error introduced by excessive closure gripping force, the cap gripping pressure, or “chuck pressure” was set to 35 psi.

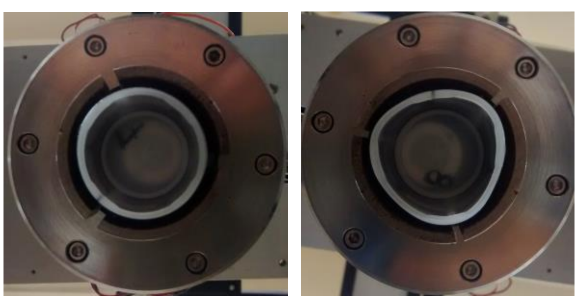

Figure 10. Closure Gripping at 35 PSI (Left) and 80 PSI (Right)

<u>Bottle Clamping Pressure:</u>
The preform clamping pressure was set to 35 psi.

<u>Downward Pressure on the Closure:</u>
The torque-constant and angle-constant application methods used in this study caused ~¼” vertical travel of the closure. To minimize the top-load introduced friction between the closure and bottle threads during testing, a floating chuck was utilized (Fig. 11).

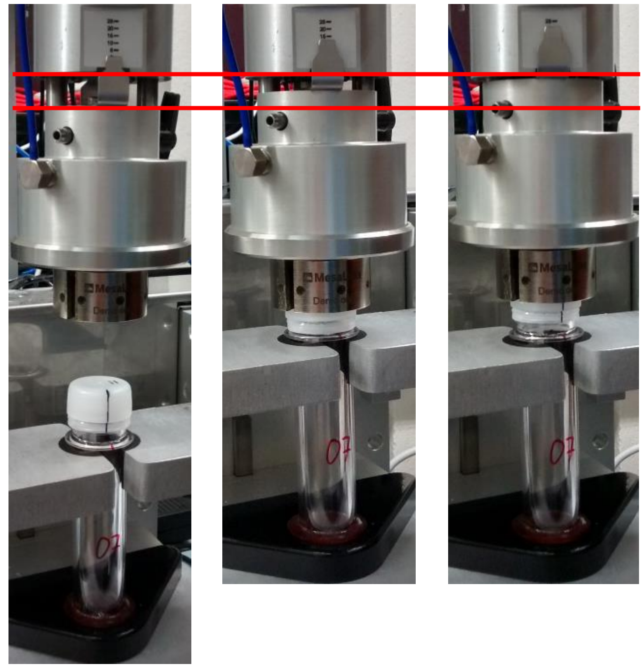

Figure 11. Floating Chuck

<u>Torque-Testing Sample Preparation</u>

The thread start on the closures was manually marked (Fig. 12, left) The preform thread start was indicated by a cutout on the preform which was then manually marked for additional visibility. The repeatability of the manually applied closure and preform thread alignment was approximately +/- 5 degrees.

Figure 12. Thread Reference Points and Alignment

<u>Torque-Testing</u>

A total of 56 closures were tightened for each of the three materials. Twenty-eight closures were applied at 20 in.lbs for each material to evaluate torque-constant application effects. Twenty-eight closures for each material were tightened to 630 degrees for evaluation of angle-constant application effects. Of the 28 samples, the closures were removed at controlled intervals (dwell time) of 15 minutes (D0.01), 1 day (D1), 7 days (D7) and 14 days (D14). The application torque was recorded for the samples applied at a constant application angle, and the resulting application angle was recorded for the samples applied at a constant application torque. The removal torque was measured for each sample.

An additional 28 samples with no slip were prepared at a constant application angle of 520 degrees for further study.

All results were analyzed in Excel and Minitab.

Results

Due to the combination of low molecular weight and high molecular weight polymer fractions, bimodal HDPE resins have a broader molecular weight distribution (MWD) at the same melt index and density compared to monomodal resins. The increased MWD of bimodal HDPE resins allows for greater shear thinning during high-speed closure injection molding, and therefore improved processability compared to monomodal HDPE at the same MI [5]. Due to the greater entanglements [6] from the incorporated high molecular weight phase [7], bimodal resin tends to relax less on the neck finish than those with lesser molecular entanglements (i.e. lower ESCR) at the same MI and density.

Bimodal closures maintain greater dimensional stability over time once applied to a neck finish, due to their increased ability to resist the stresses induced by the pressure exerted by the interaction of the closure skirt with the bottle neck finish following application. This greater dimensional stability results in reduced risk of leakage due to relaxation (creep) of the closure's contact with the neck finish caused by other external factors such as excess pressure from the bottle contents, impact, etc. resulting in loss of seal.

The no slip samples illustrate the effect of polymer relaxation over time, a significant portion of which occurs in the first 24 hours following application (Fig. 13).

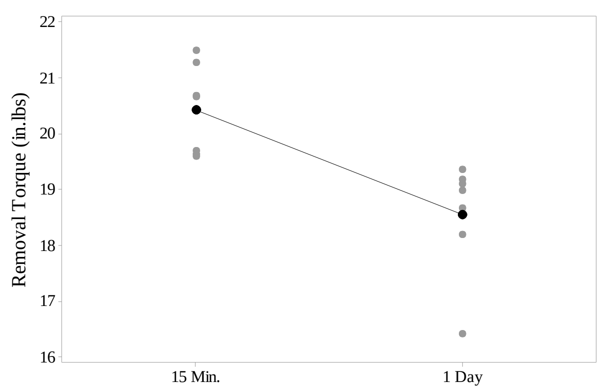

Figure 13. Removal Torque with Mean Connecting Line: Angle-Constant Application at 630° for No Slip Sample Set at Removal Time of 15 Minutes and 1 Day Following Application

It was therefore determined that, in order to get a good "initial" reading for the time-series study, a sample set for each material type and application method should be tested within 15 minutes following application to the neck.

Torque-Constant Application Analysis

When closures were applied at a constant torque of 20 in.lbs, the closures containing low and standard slip consistently terminated at an application angle of approximately 630° (Fig. 14).

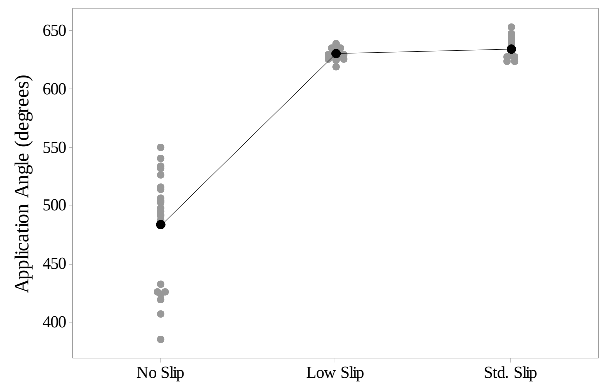

Figure 14. Resulting Application Angle from Torque-Constant Application at 20 in.lbs for No, Low and Standard Slip Samples with Mean Connecting Line

This is due in part to the presence of a slip layer on the skirt of the closure sufficient to avoid the blocking effect of the friction resulting from the interaction of the HDPE closure and PET neck finish surfaces during application. For the two slip containing sample sets, the low application angle variation is a result of the PET neck contacting the closure ceiling next to the pressure block seal (Fig. 15), making it impossible to screw the cap at a more advanced angle.

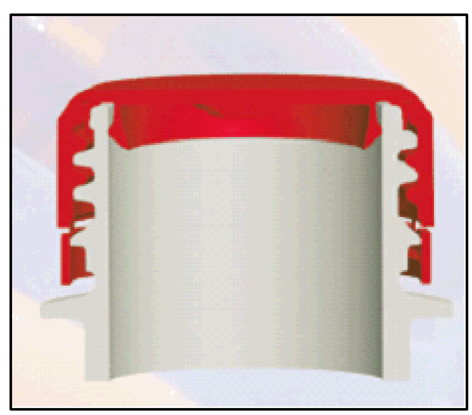

Figure 15. Closure Screwed at Max Angle, PET Neck in Place Against the Closure Ceiling

If the capping head torque value is set such that it over-screws the closure above 630°, the closure will then strip

(Fig. 16) leading to seal integrity failure. Over-torqueing closures is a common problem with lighter, thin-walled closures.

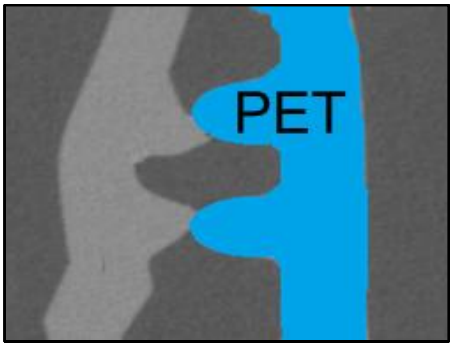

Figure 16. Closure Skirt Expansion Resulting from Over-Torqueing

Table 3: Removal Torque: Torque-Constant Application

	No Slip	Low Slip	Std. Slip
15 Min. (D0.01)	12.7 in.lbs	17.7 in.lbs	17.3 in.lbs
1 Day (D1)	12.4 in.lbs	14.7 in.lbs	14.1 in.lbs
7 Days (D7)	11.6 in.lbs	14.0 in.lbs	13.1 in.lbs

For the closure set containing no slip, the high variation and lower value of the application angle results are due to the frictional blocking effect of the HDPE on PET. Minute variability in the individual closure and preform dimensions can also play a role in the resulting disparity between application angle results for the no slip samples applied under constant torque.

The no slip samples studied gave torque removal values ranging from 11.6 in.lbs to 12.7 in.lbs over the testing duration:

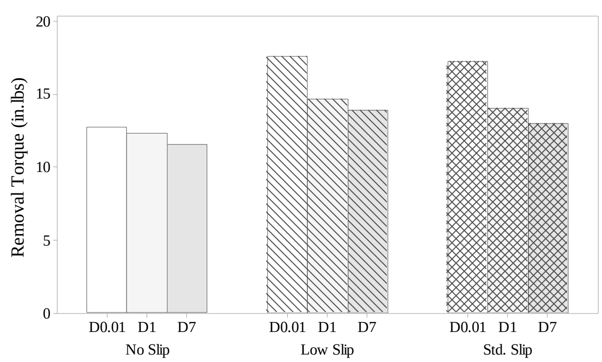

Figure 17. Removal Torque after 15 Minutes (D0.01), 1 Day (D1) and 7 Days (D7) for Torque-Constant Application at 20 in-lbs for No, Low and Standard Slip

The low removal torque values for the no slip samples as compared to the low and standard slip samples are attributed to the lower initial application angle of the no slip samples.

When comparing the low and standard slip data sets, the standard slip gives comparable, but lower overall removal torque values at each test time interval and slightly greater differences between time intervals compared to the low slip.

In these results we see not only the effect of the initial relaxation of the polymer, but also the effect of slip blooming over time on the removal torque.

Angle-Constant Application Analysis

The trend of lower mean application torque (Fig. 18) required when applying caps to a constant angle for standard versus low and no slip is to be expected as the initial blooming of the slip agent would cause the surface of the closure coming in contact with the PET neck finish to have a reduced coefficient of friction, therefore requiring less force to overcome the lower frictional forces encountered during closure application.

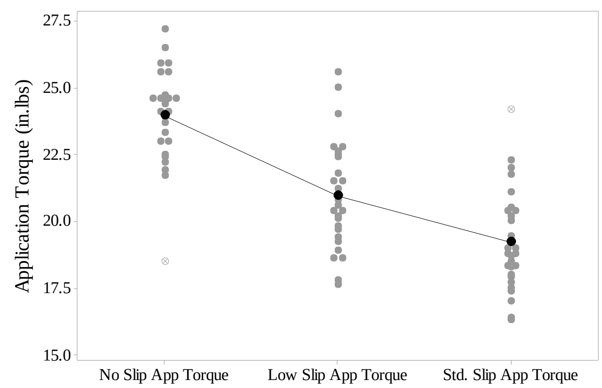

Figure 18. Resulting Application Torque from Angle-Constant Application at 630° for No, Low and Standard Slip with Mean Connecting Line

Notably, to apply the closures to the angle of 630°, the no slip sample set required a mean application torque of ~24 in.lbs to overcome the opposing frictional forces encountered during application. This results in a very effective seal to prevent leaks, however, the closures become difficult to-open or are un-openable, even after relaxation of the polymer when torque removal measurements are taken a week later (Fig 19).

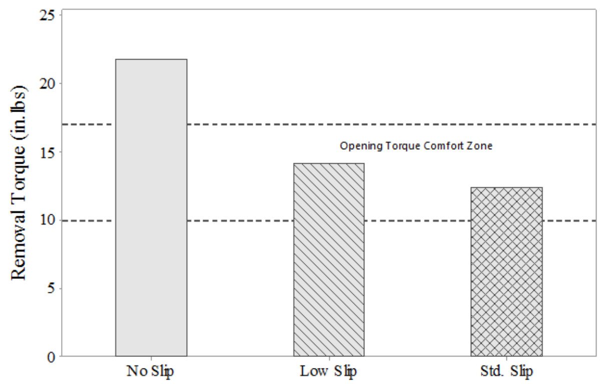

Figure 19. Removal Torque after 7 Days for Angle-Constant Application at 630° for No, Low and Standard Slip

The "opening torque comfort zone" for users is typically a removal value of between 10 and 17 in.lbs required opening force.

When the angle was held constant during closure application, greater variation was observed in the torque removal values overall for each slip level at each tested time interval.

Conclusions

For a given closure design and material, identifying the ideal application method is critical to provide acceptable, repeatable and in-control torque removal values. In this study both torque-constant and angle-constant application methods were evaluated over time, using materials with variable slip concentration to gain more insight on the impact of minor material changes on closure application.

For torque-constant and angle-constant applications, both low and standard slip samples gave torque removal values within the opening torque comfort zone following the first 24 hours after closure application. The outcome of this study shows that while there was not a significant difference between the torque required for the application and removal of the closures containing low and standard slip as compared to the samples containing no slip, overall the standard slip samples required lower application and removal torques than the low slip samples:

Table 4: 14 Day Removal Torque

	Low Slip	Std. Slip
Angle-Constant (630°)	14.4 in.lbs	12.8 in.lbs
Torque-Constant (20 in.lbs)	13.4 in.lbs	13.6 in.lbs

The role of application angle on resulting torque removal values is best seen when comparing the no slip sample results:

Table 5: 14 Day Removal Torque; No Slip

	Application Angle	Application Torque	Removal Torque
Angle-Constant (630°)	630°	22.8 in.lbs	16.7 in.lbs
Angle-Constant (520°)	520°	22.8 in.lbs	12.5 in.lbs
Torque-Constant (20 in.lbs)	477°	20.0 in.lbs	10.3 in.lbs

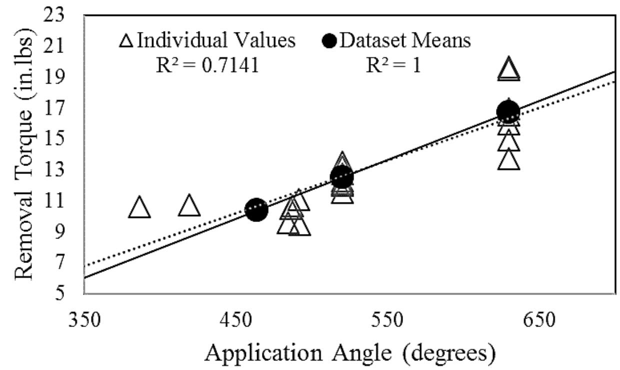

Figure 20. 14-Day Removal Torque vs. Application Angle with R-Squared Value for Individual Values [Dotted Trendline] and Dataset Means [Solid Trendline] (See Table 5)

For the tested closure design, the absence of a slip agent greatly impacted the consistency of application angle and torque and therefore removal of the closure. For the no slip samples, as application angle increased, the required application torque correspondingly increased (Fig. 20).

Notably, in this study for the no slip samples, removal torque showed better correlation with application angle than with application torque as can be seen in the R-squared values shown in Figures 20 and 21. The relationship between application angle and torque removal was also evident for the 7-day, 1-day and 15 minute samples as compared to application torque versus torque removal.

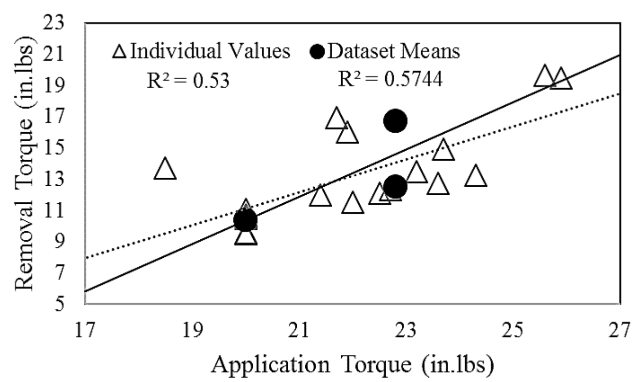

Figure 21. 14-Day Removal Torque vs. Application Torque with R-Squared Value for Individual Values

[Dotted Trendline] and Dataset Means [Solid Trendline] (See Table 5)

For the angle-constant application at 630 degrees, the resulting removal torque after 14 days at ~16.7 in.lbs indicates a good seal for the product has been achieved, however, the removal torque is at the upper end of the user's opening torque comfort zone, and may be ergonomically unsuitable for all beverage consumers. For torque-constant application at 20 in.lbs (mean application angle of ~477°), the removal torque dropped after 14 days to ~10.3 in.lbs. At this low removal torque, the opening torque is at the bottom of the user's comfort zone, however, there is the risk that sufficient seal is not consistent throughout production.

Making small changes to an existing resin for a closure, such as adding, removing, or adjusting slip levels greatly impacts the final part performance. In the same way, adjustments to a resin's melt flow properties, density, or modality will also alter performance on a capping line. Individual resins vary in their tolerances for the minimum and maximum application torque necessary to ensure good seal and comfortable torque removal. In the same way, the material composition can greatly affect the range of accepted application angles in non-destructive visual seal integrity testing. In order to capture the most value, a thorough understanding of the closure material, closure design and closure application tolerances are vital for bottlers to be able to minimize defects and maximize product quality.

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