GPEC 2004 Paper Abstract #52:

Title: **Cesa-extend a User Friendly Technology to Enhance Reprocessing and Recycling of Condensation Plastics**

Author(s): V. Karayan, Clariant Masterbatches, and M. Villalobos, Johnson Polymer

Recycling of condensation thermoplastics such as polyesters (PET, PBT), polyamides (6, 6-6), polyurethanes, polycarbonates, and their blends, has found severe limitations owing to a simple reason: the costs associated to current process technology employed to revert MW degradation of these thermoplastics renders the recycled products uneconomical and/or unsuitable for many demanding applications. As a result, degraded post-consumer reclaimed plastics is still the main recycle stream, mostly directed to low value added applications such as fibers and film.

During 2003, Clariant Masterbatches and Johnson Polymer jointly introduced to the market a family of chain extenders or "recycling aids" under the trademark Cesa-extend. These additives are based on proprietary technology of multi-functional acrylic oligomers formulated into masterbatches tailored for effective and user-friendly use in different thermoplastic systems. Cesa-extend products are characterized by their ability to dramatically increase the molecular weight, as well as the mechanical and rheological properties of virgin, reprocessed, and post-consumer recycled condensation plastics when used in very low concentration in simple extrusion or injection molding equipment.

Multiple examples in which recycled feedstock has been enhanced with Cesa-extend products during a simple extrusion step to meet demanding engineering applications requirements will be given in the areas of polyesters, polyamides and other thermoplastics.

Vahe Karayan PhD
Technical manager
Clariant Additive Masterbatches
382 Arbor Court
Winchester, VA 22602
PHONE: 540-665-1865 X 106
FAX: 540-665-2917

CESA®-extend a New Technology to Enhance Reprocessing and Recycling of Condensation Polymers

Vahe Karayan PhD, Clariant Additive Masterbatches, Marco Villalobos PhD, Johnson Polymers

INTRODUCTION:

Polyethylene terephthalate (PET) is an important commercial polymer used widely in the manufacture of plastic products such as films, bottles, sheet and other molded and extruded articles. In a life cycle, these materials experience several process steps, from a synthesis process, followed by an extrusion step, and a final processing step which may be another compounding extrusion operation followed by thermoforming, blow molding, fiber spinning, or injection molding in the molten state. All of these steps occur under high temperature conditions which affect the molecular weight (MW), rheological properties, melt strength and processing abilities. As a result recycled PET is mostly employed in low-end applications or is reconstituted by a solid stating process that requires a very large capital investment.

The current yearly consumption of recycled PET in the United States is about 700 million pounds. In recent years, increased attention has been focused on improved methods of recycling the articles made from these polymers, with an eye toward resource conservation and environmental protection.

CURRENT TECHNOLOGY:

There are a number of processes in the industry used to minimize loss in molecular weight and maintain or even increase the molecular weight of the PET for processing or recycling. Most of these processes employ as main processing equipment either an extruder, a solid state polycondensation reactor, or both in sequence, or similar equipment designed for melt or high viscosity material processing.

As a critical component of any of these processes, chemical reactants known in the industry as "chain extenders" are employed. Chain extenders are, for the most part, functional molecules that during any or all of the described processing steps are added as additives to the extruder or reactor with the purpose of "re-coupling" polycondensate chains that have depolymerized to some degree. Normally the chain extender has two or more chemical groups that are reactive to the chemical groups formed during the molecular weight degradation process. By reacting the chain extender molecule to two or more polycondensate fragments it is possible to re-couple them thus decreasing or even reverting the molecular weight degradation process.

In order to have efficient chain extension at reasonable residence times most of the known chain extenders require the use of pre-dried polycondensate material, operation at high vacuum, and varying amounts of catalyst and stabilizers, to be employed during processing. Without these features the extent of molecular weight increase is limited and the resulting product shows lower molecular weight and less than desired properties.

As the functionality of the chain extender increases, so does the number of polycondensate chains that can be coupled onto each chain extender molecule, and thus its effectiveness in re-building molecular weight. However, it is easy to see that as the functionality of these chain extenders increase so does the degree of branching of the resulting product and the potential for onset of gelation. The strong negative effects that extensive branching has on the degree of crystallinity and thus on the mechanical properties of a semi-crystalline polycondensate, as well as the negative implications of the presence of varying amounts of gel in any product.

A need exists for chain extenders that may be used in any suitable process while avoiding the processing limitations described above. Such chain extenders provide substantial economic advantage in processing, reprocessing and recycling of polycondensates over existing chain extenders and the methods for their use.

CESA®-extend a MULTIFUNCTIONAL OLIGOMERIC CHAIN EXTENDER:

One type of chain extender that has been effective in overcoming the problems encountered are those based on epoxy-functional styrene acrylic copolymers produced from monomers of at least one epoxy-functional acrylic monomer and at least one non-functional styrenic and/or acrylate monomer. Clariant Additive Masterbatches and Johnson Polymers Company have introduced a multifunctional oligomeric chain extender masterbatch, CESA®-extend, to reduce the degradation of PET due to process heat and moisture.

This product is particularly well suited for use with reprocessed or recycled plastics. It is characterized by its ability to revert the post-processing molecular weight decrease in different polycondensates from the minimum value reached without chain extension, back to the initial molecular weight values or even larger than the original molecular weight values. This is achieved without the incidence of gel and without adverse effects on mechanical, thermal, or rheological properties at a target polycondensate molecular weight.

CESA®-extend provides several benefits in a variety of applications. CESA®-extend can be used as a substitute for solid state polymerization in recycling of PET. Compounders can benefit from improved compatibility with other types of plastics such as other polyesters, polycarbonate, polyamide, etc. Converters can benefit from improved extrusion blow molding and melt strength, injection blow-molding crystallinity and toughness in blow molding operations. In foamed sheet, benefits can be expected due to countered effects of endothermic foaming agents. In industrial fabrics the use of these chain extenders can improve coating adhesion, tenacity and melt strength. The following data will show the influence of the use of CESA®-extend on the melt strength and processability of recycled PET.

OBJECTIVE:

A series of laboratory experiments were conducted to determine the effect of the addition of CESA®-extend on the die sag and processability of PET extruded sheet.

EQUIPMENT:

A 27 mm Leistritz twin screw extruder with an 8" sheet die fitting and down stream pick up rolls were used for the experiments.

The pick up rolls were aligned to the same height as the extruder die lip, and the distance between the two was kept constant at 6.25 inches apart. This distance was determined by extruding a 100% virgin PET and the process was stabilized without tearing the sheet or approaching the die lip so close that the sag was eliminated.

The distance between the die lip and the floor was measured as 41.25 inches, the RPM of the extruder and the speed of the pick up rolls were kept constant at 90 RPM and 4.3 ft/min respectively. The melt temperatures were also kept constant at 453°F, torque and discharge pressures were recorded and reported for each experiment (table 1).

EXPERIMENTS:

Once these reference conditions were set, a series of extrusions were run with the addition of two different CESA®-extend masterbatches, one was a masterbatch in a PET carrier (CESA®-extend 9930), and the other was a styrenic carrier masterbatch (CESA®-extend 1598). After each run was stabilized the distance between the floor and the lowest sag point in the film between the die lip and the pick up rolls was measured and recorded (table 2). Each test sample was dried at 200°F for six hours and the moisture contents were measured to be below 100 ppm.

Table 1:

Sample	Description	% Torque	Discharge Pressure PSI
Sample 1	100% PET	55	70
Sample 2	0.25% CESA-extend 9930	49	90
Sample 3	0.50% CESA-extend 9930	44	100
Sample 4	1.00% CESA-extend 9930	47	120
Sample 5	2.00% CESA-extend 9930	47	170
Sample 6	0.25% CESA-extend 1598	40	80
Sample 7	0.50% CESA-extend 1598	38	100
Sample 8	1.00% CESA-extend 1598	36	120
Sample 9	2.00% CESA-extend 1598	40	180

Table 2:

Sample	Description	Sheet To Floor (inches)	Sag (inches)	Die Sag Reduction
Sample 1	100% PET	39.5	1.75	-
Sample 2	0.25% CESA-extend 9930	39.5	1.75	0.00%
Sample 3	0.5% CESA-extend 9930	39.75	1.5	14.29%
Sample 4	1.00% CESA-extend 9930	40.25	1	42.86%
Sample 5	2.00% CESA-extend 9930	41.25	0	100.00%
Sample 6	0.25% CESA-extend 1598	39.75	1.5	14.29%
Sample 7	0.50% CESA-extend 1598	40	1.25	28.57%
Sample 8	1.00% CESA-extend 1598	40.5	0.75	57.14%
Sample 9	2.00% CESA-extend 1598	41.25	0	100.00%

OBSERVATIONS & CONCLUSIONS:

The results in tables 1 and 2 show that the addition of either of the CESA®-extend masterbatches slightly reduces torque, and increases die pressure , resulting in improved and eventual elimination of the sag observed when no

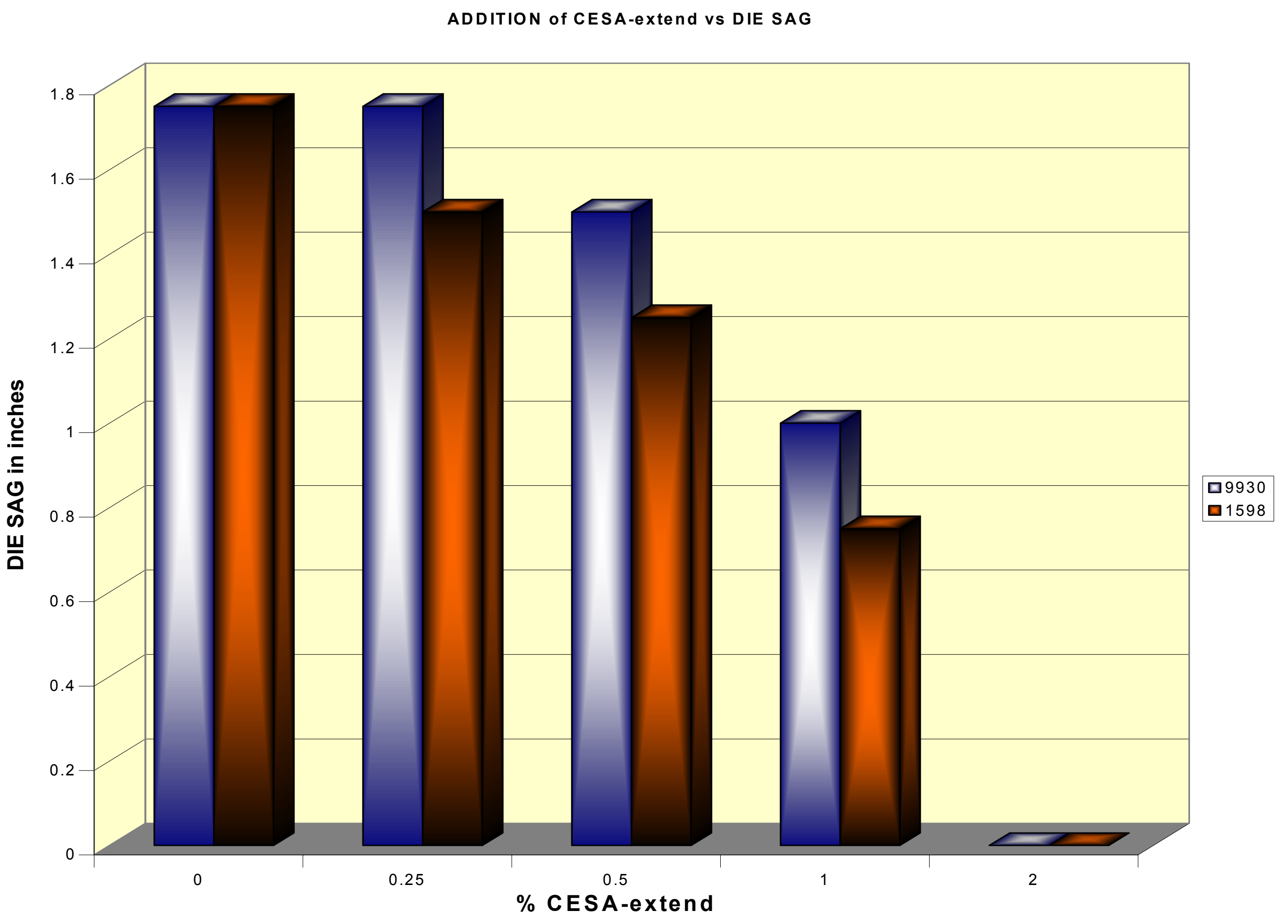

Figure 1:

CESA®-extend has been added to the PET (Photo 1 & 2). The CESA®-extend with the styrenic masterbatch is slightly more effective compared to the PET carrier based masterbatch, at 0.25% of either carrier masterbatch shows no effect in the case of the PET carrier masterbatch, compared to 14.29% less sag with the styrenic carrier masterbatch (fig. 1 & 2).

The virtual elimination of the severe die lip sag demonstrates the positive effect of CESA®-extend on the melt strength and dramatic improvement in processing capability of PET sheet.

Figure 2:

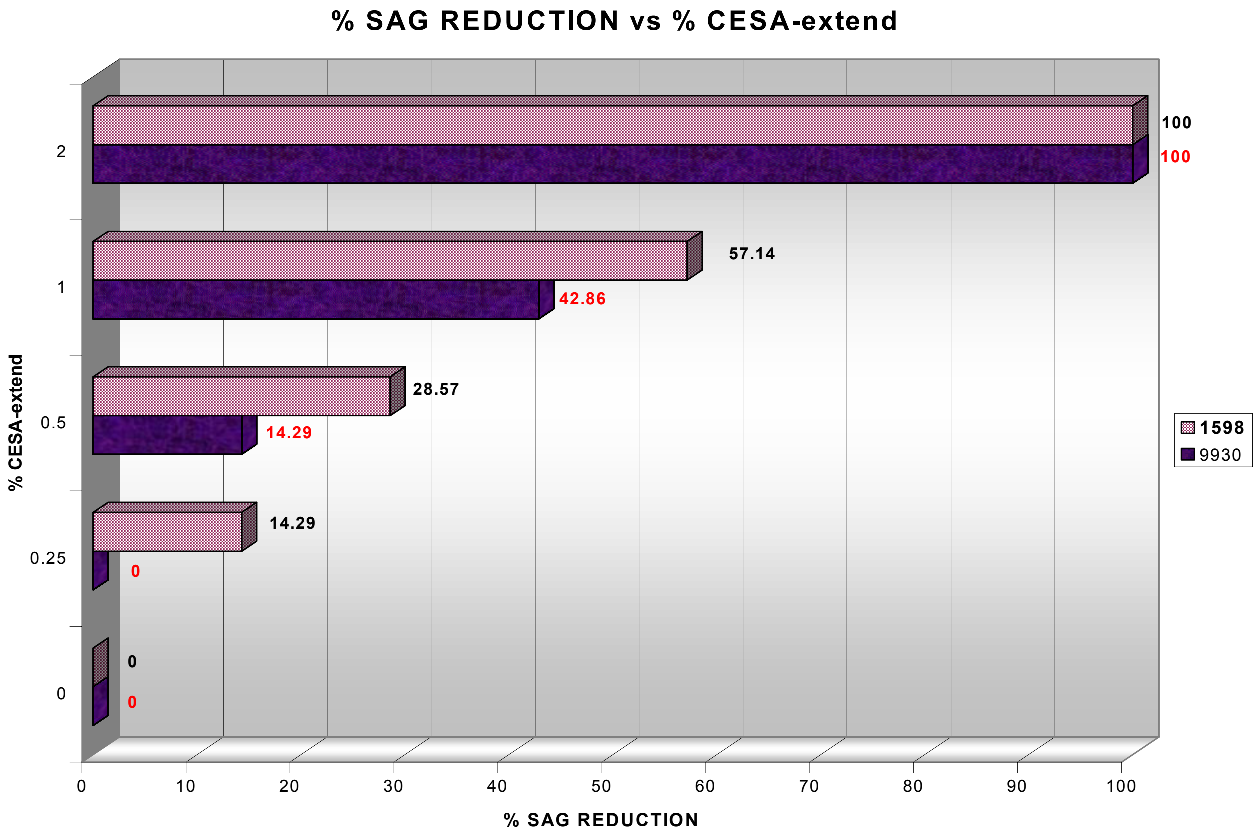

Photo 1:

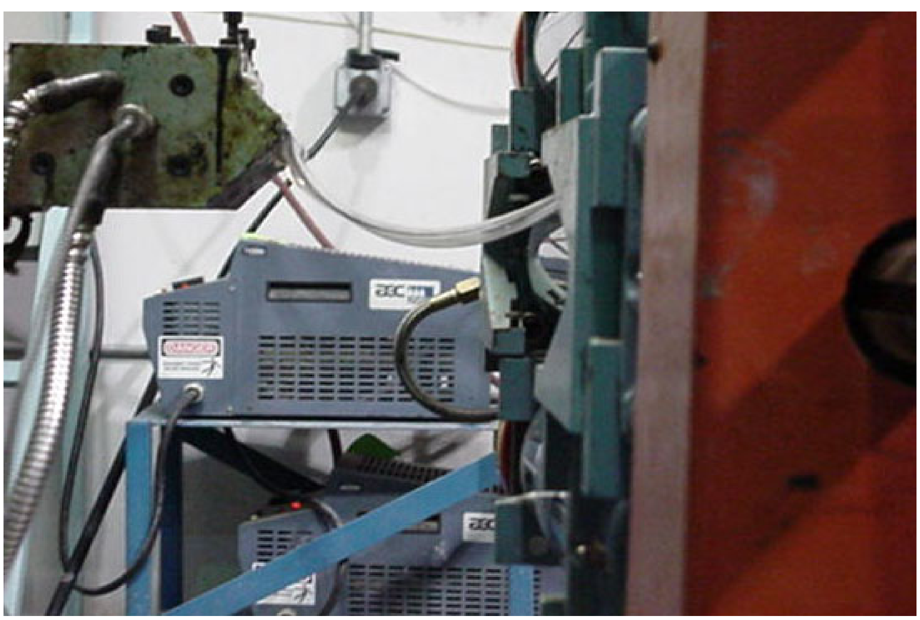

Photo 2: