Processing of non-dried PET-Bottle-Scrap using Co-rotating Twin Screw Extruders

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PET consumption has been recording above-average growth rates for many years. The main reasons for this growth are the increasing popularity of PET for non-returnable and returnable bottles for beverages as well as the proliferation of various types of PET film packs, for example as deep-freeze packs suitable for oven or microwave use.

An increasing volume of reclaim material is accompanying this growth. Most of the scrap produced during processing – edge trim or punch scrap from the thermoforming of film packs, for example – is treated and recycled in-house, but large quantities of washed, sorted regrind material are also coming out of the consumer cycle.

PET being a polycondensate is hygroscopic and very quickly absorbs the equilibrium moisture of 1000-3300 ppm water (depending on the particular product). Therefore during plasticising non dried product, hydrolytic degradation will start immediately, as it is 10,000 times faster than thermal degradation caused by excessively high processing temperatures and 5,000 times faster than thermo-oxidative degradation caused by the presence of oxygen. Therefore the removal of retained moisture is the main problem for the processing and compounding of PET, even for virgin material. There are several possibilities for achieving low process moisture content:

- Standard-crystallization at 150°C and drying at 150–180°C
- Air-drying process at temperatures of up to 60 °C without crystallization
- Combination of pre-drying or conditioning and vacuum degassing
- Vacuum degassing during extrusion

This presentation will review the Co-rotating twin-screw extrusion technology which eliminates not only the pre-drying of the recycled or virgin material but also the crystallizing and agglomerating process. With over 65 lines in operation, the technology has been shown to be suitable for virgin material, for all kinds of regrind and recyclate, and for blends of these materials. In addition to pellets it can directly produce sheet, film and fiber.
Improvements and new developments in the Processing of undried PET-Bottle-Flakes on Co-rotating Twin Screw Extruders

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Dr. Paul Andersen

1. Introduction

The consumption of PET has been recording above-average growth rates for many years: as far as applications in the packaging sector are concerned, the rise has been around 9% per year. The main reasons for this growth are the increasing popularity of PET for non-returnable and returnable bottles for beverages as well as the spread of various types of PET film packs, for example as deep-fryeze packs suitable for oven or microwave use.

A mounting volume of reclaim material from various sources is accompanying this growth. Most of the scrap produced during processing – edge trim or punch scrap from the thermoforming of film packs, for example – is treated and recycled in-house, but large quantities of washed, sorted regrind material are also coming out of the consumer cycle, and are being traded as recyclate for subsequent processing.

Even with virgin material, pre-drying is of eminent importance in the conventional processing of PET on single screw extruders. PET being a polycondensate is hygroscopic and very quickly absorbs the equilibrium moisture of 1000-3300 ppm water (depending on the particular product).
Fig. 1: Polycondensation of PET

While plastifying undried product, hydrolytic degradation will start immediately, as it is 10,000 times faster than thermal degradation caused by excessively high processing temperatures and 5,000 times faster than thermo-oxidative degradation caused by the presence of oxygen. Therefore the removal of retained moisture is the main problem for the processing and compounding of PET. There are several possibilities for achieving low process moisture content:

- Standard-crystallization at 150°C and drying at 150–180°C
- Air-drying process at temperatures of up to 60°C without crystallization
- Combination of pre-drying or conditioning and vacuum degassing
- **Vacuum degassing during extrusion**

The preparation of the feedstock is even more complex in the case of recycled material. It has to be agglomerated, crystallized and pre-dried before extrusion and, apart from that, it also has to be sorted according to its intrinsic viscosity (iV), which differentiates one material from another. It is the iV that governs which field of application the PET recylcate can be used in.

<table>
<thead>
<tr>
<th>Viscosity</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.85 – 1.5 dl/g</td>
<td>Monofilaments, Technical yarns, High-strength packing straps</td>
</tr>
<tr>
<td>0.80 – 0.84 dl/g</td>
<td>Bottles for food (Mineral water, Softdrinks, Beer) and other non-food applications</td>
</tr>
<tr>
<td>0.68 – 0.76 dl/g</td>
<td>A-PET-Films and Sheets for Thermoforming</td>
</tr>
<tr>
<td>ca. 0.6 dl/g</td>
<td>Bi-axial oriented films</td>
</tr>
<tr>
<td>0.58 – 0.62 dl/g</td>
<td>Staple fibers (Chopped fibers), Filling fibers, Carpet fibers, Spin bonded fabric</td>
</tr>
<tr>
<td>0.60 – 0.80 dl/g</td>
<td>Reinforced materials, Polymer blends</td>
</tr>
</tbody>
</table>

Table 1: Typical intrinsic viscosities and fields of application
2. Twin screw process

For the preparation and direct extrusion of PET, Coperion Werner & Pfleiderer has developed a technology, which eliminates not only the pre-drying of the recyclate but also the crystallizing and agglomerating process. The company introduced this technology for the first time at K 92 and has since sold more than 60 units for different applications. Over the years, the process engineering has been constantly refined and adapted to the needs of the market. This direct process is based on the good processing properties and the high venting efficiency of the twin screw compounder ZSK MEGAcompounderPLUS (McPLUS).

Fig. 2: ZSK MEGAcompounderPLUS with segmented barrels and screw elements

The technology is suitable for virgin material, for all kinds of regrind and recyclate, and for blends of these materials, and can directly produce film, fiber or injection molding granules. The following table shows a comparison, drawn up externally, of the ZSK process, with various single screw processes. It highlights the many benefits of processing in a single step.

- The energy and time savings, because there is no need for pre-drying or crystallization.
- The very high quality of the end product, because there is only a minimum decrease in the iV – just a few percent – during processing and because glass-clear material has a much lower yellow cast than with processes involving pre-drying.
The high flexibility, because, due to the good self-wiping characteristics of the twin screws, changing a formulation and color takes less than 30 minutes.

The much simplified logistics, because granulated virgin material and different reclaim material (regrind, agglomerates, flakes) can be processed together even if the iV is different.

With the ZSK process, the throughput is limited neither by the pre-drying capacity nor by the screw size. For example, a ZSK McPLUS with a screw diameter of D = 92 mm achieves a throughput rate of 2,000 kg/h. For this throughput at least a 200 mm single screw would be necessary.

3. Venting is the Decisive Factor for quality

Figure 3 shows the typical structure of a twin screw compounder for the processing and direct extrusion of PET. The gravimetric metering systems that are designed for the relevant components introduce the materials into the feed opening. 100% virgin material, 100% reclaim material or mixtures of the two can be used in any ratio. The only requirement made on the reclaim material concerns its purity. It can contain a maximum of 50 ppm PVC and 50 ppm Polyolefins, because these materials degrade at the melt temperature of 270-280°C that is needed for PET, with the result that decomposition products contaminate the PET and – especially in the case of PVC – cause more intense degradation of the PET. Fiber waste can also be used if it has been shredded in a compactor and condensed to produce a free-flowing, meterable bulk material. The processing section of the compounding extruder comprises 8
barrel sections, each 4D long, giving a total length of 32D. The screw is designed with appropriate screw elements specifically for the particular function - melting, venting, homogenizing.

In the third barrel section, in other words immediately after commencement of the melting process, a large proportion of the water released during heating, escapes by atmospheric venting. Subsequently, a vacuum venting system with an absolute pressure of 10 mbar max extends over barrel sections 5-7. During this period, not only is the residual moisture removed from the melt, but also any low molecular weight component that is still present or has been newly generated through material decomposition.

Fig. 4: Degassing of polymer melt
The decisive factors for the good venting efficiency are, apart from the length of the venting zone, the large melt surface area through the partial filling and the constant regeneration of the surface due to intensive mixing. Because of the large free volume and the diameter ratio of $D_0/D_i = 1.55$, the ZSK MEGAcompounder PLUS is also very suitable in this respect.

![Diagram of venting system](image)

**Fig. 5:** A side vent prevents deposits getting into melt

To extract the gas, it is advisable to use a side venting system. With this design, no deposits from the vents can get back into the melt and contaminate it. The key role played by venting in the processing of PET comes from the fact that this plastic is produced by Polycondensation, in other words through the splitting off of water.

Accordingly, at elevated temperatures and in the presence of water, the reverse reaction can take place, hydrolysis, which can break down the material. Because these are equilibrium reactions, the iV and thus the quality of the end product can be influenced solely by the choice of process conditions and without any addition whatsoever of coupling agents.
Generally speaking, a high mechanical stress on the melt and a poor venting system encourage the degradation process. The more accurate evaluation in Fig 6 shows that the lower the standard throughput, the lower the degradation. In concrete terms, this means that a long residence time and a low filling level produce a smaller reduction in the intrinsic viscosity and, in an extreme case, can even result in a slight increase.

When recycling bottle regrind for fiber production, it is possible, during processing, to specifically degrade the PET via suitable process parameters because the starting iV value of 0.78 to 0.8 dl/g of the bottle materials is much higher that the value needed for fiber material, approximately 0.6 dl/g.
4. Discharge section and requirements to the process

The discharge section of the compounder and the downstream units must, of course, be suitably adapted, as is demonstrated in Fig. 7 for direct flat film extrusion.

Because of the low melt viscosity of the PET, a gear pump is always used to build up the extrusion pressure. With its high efficiency in building up pressure, it makes an important contribution to keeping the melt temperature down. A subsequent filter with an automatic screen changer catches any remaining impurities. The fineness of the screen will depend on the specifications for the manufacture of the end production of fibers and biaxial oriented film. The filtered melt is extruded with a slit die or added to the subsequent spinning process via spinning pumps.
The concept described here is also suitable for multi-layer films as is shown by the successful operation of a film production line for the manufacture of a coextruded three-layer film at a well-known Danish packaging producer. One line in the facility has a ZSK 92 Mc as the main extruder and two ZSK 50 Mc’s as coextruders. The other line has a ZSK 119 McPLUS with a ZSK 70 McPLUS as co-extruder. The prime reasons for choosing this concept were the energy-savings due to the elimination of the pre-drying process, the very short changeover time of only 30 minutes for a material color change, and the high mechanical and optical quality of the film.

5. Bottle Recycling

The table below shows part of the results that have been achieved with different raw materials, different residual moisture contents and 5 mbar absolute vacuum. It is obvious that materials of iV around 0.6 dl/g show essentially no degradation whereas the higher viscosity materials like the 9921W degrade about 0.03-0.04 dl/g. Only the bottle flakes with a comparable viscosity of 0.08 show a higher degradation at constant operating conditions.

<table>
<thead>
<tr>
<th>Raw Materials</th>
<th>IV</th>
<th>Moist.</th>
<th>Film IV</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Film Regrind</td>
<td>0.66</td>
<td>677</td>
<td>0.65</td>
<td></td>
</tr>
<tr>
<td>Amorphous pellets</td>
<td>0.66</td>
<td>496</td>
<td>0.66</td>
<td></td>
</tr>
<tr>
<td>30% Pellets crystallized + 70% Film Regrind</td>
<td>0.6+0.64</td>
<td>...</td>
<td>0.67</td>
<td></td>
</tr>
<tr>
<td>Film Regrind</td>
<td>0.64</td>
<td>2279</td>
<td>0.65</td>
<td></td>
</tr>
<tr>
<td>Film Regrind</td>
<td>0.64</td>
<td>2279</td>
<td>0.57</td>
<td>no vacuum</td>
</tr>
<tr>
<td>PET-G virgin</td>
<td>0.8</td>
<td>518</td>
<td>0.77</td>
<td></td>
</tr>
<tr>
<td>PET-G flakes</td>
<td>0.72</td>
<td>2815</td>
<td>0.71</td>
<td></td>
</tr>
<tr>
<td>85% PET-G virgin + 15% PET-G flakes</td>
<td>0.8+0.72</td>
<td>...</td>
<td>0.76</td>
<td></td>
</tr>
<tr>
<td>Bottle Flakes, partially crystallized</td>
<td>0.8</td>
<td>2640</td>
<td>0.72</td>
<td></td>
</tr>
<tr>
<td>50% Pellets crystallized + 50% Bottle Flakes</td>
<td>0.78+0.8</td>
<td>...</td>
<td>0.72</td>
<td></td>
</tr>
<tr>
<td>Preform-Flakes bag 5</td>
<td>0.8</td>
<td>2187</td>
<td>0.73</td>
<td></td>
</tr>
<tr>
<td>Preform-Flakes bag 6</td>
<td>0.8</td>
<td>2486</td>
<td>0.72</td>
<td></td>
</tr>
<tr>
<td>Bottle Flakes</td>
<td>0.78</td>
<td>6727</td>
<td>0.69</td>
<td></td>
</tr>
<tr>
<td>&quot; dried</td>
<td>0.78</td>
<td>1010</td>
<td>0.70</td>
<td></td>
</tr>
<tr>
<td>Pellets crystallized 9921W</td>
<td>0.81</td>
<td>359</td>
<td>0.77</td>
<td></td>
</tr>
<tr>
<td>Pellets crystallized 1</td>
<td>0.8</td>
<td>240</td>
<td>0.77</td>
<td></td>
</tr>
<tr>
<td>Pellets crystallized 2</td>
<td>0.8</td>
<td>468</td>
<td>0.76</td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Test Results of the trials carried out on ZSK 40McPLUS (IV in dl/g, residual moisture in ppm)

To find out the reason for the difference between bottle flakes and virgin material one has to look at the sources.
### Quality differs dependant on bottle collecting systems

#### Decreasing flake quality:
- Selection of the bottles from the refillable bottles cycle
- Bottles from the returnable deposit system (Germany)
- Material separated from bottle collection
- Unsorted plastic waste from domestic, shops and industry

#### Contamination:
- Coloured PET-Bottles (green, blue, brown)
- Prints
- Different Polymers like PE, PP, PVC, Polyamide, PVAC (Barrier layers, caps)
- Metals, Glue, Sealing materials
- Syrup, Aroma and taste additives
- Paper, cellulose
- Oxygen absorbers, Iron, Kaoline
- Large particles and foreign bodies like sand, Glass fibers, Fuel residuals,
  Mineral Oil, Household chemicals

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Table 3: Quality Requirements and possible contaminations for bottle flakes

Some of those contaminates can be separated in an appropriate melt filter, others like e.g. glue or sealing materials, foreign plastics and residual detergents from the washing process have a direct influence on the degradation of the PET, so a proper grinding and washing process is an eminent step to achieve high quality material.

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**Fig. 8:**
Cellulose Fibers that passed the melt filtration
When processing PET bottle material for renewed bottle production (bottle-to-bottle process), the compounder is equipped with a strand pelletizer or an underwater pelletizing system after the combination of gear pump and screen changer. Bottle preforms can be injection molded directly from the resultant granules. Their use in the food segment is possible without restriction following a 12-hour solid phase post-condensation period in a solid state reactor. The US Food and Drug Administration has approved these granules for food-contact applications.

Fig. 9: Bottle to bottle recycling
6. Glycolysis

The broad processing bandwidth of twin-screw units means that it is also possible to recycle reclaim material that is heavily contaminated with fines (dust, paper). In such cases, a small amount of e.g. ethylene glycol (EG) is added during processing to bring about a specific degradation process that reduces the iV value from e.g. 0.8 dl/g to 0.3 dl/g or less. This corresponds to a reduction in the molar mass from around 75,000 to 22,000.

Fig. 10: ZSK 58 for the recycling of bottle flakes

From this very low viscosity melt, the fine impurities can be removed very effectively by filtration with fine-mesh screens (mesh size below 20 µm for the second melt filter).
The cleaned PET melt can subsequently be returned into the esterification process where it is polymerised into virgin material.

Fig. 11: Flow Diagram for the glycolysis

Fig. 12: Possible applications after the glycolysis step
7. Machine sizes

Coperion Werner & Pfleiderer has manufactured twin screw extruders for the plastics and food industry for more than 50 years. Since its introduction, the technical development of compounding on co-rotating twin screws has been decisively influenced by improvements in design and materials of construction. For example, from the first generation ZSK 83 to the MEGAcompounderPLUS series (launched in 2004), torque, speed and free volume have had a significant number of step change improvements.

The McPLUS is the appropriate twin-screw for the compounding and recycling of PET. Due to its high torque, the PET can be processed very gently at low screw rpm, due to the large free volume even high amounts of volatiles can be devolatilized safely. For the PET process the screw speed of the McPLUS is limited to a speed of 400 rpm although for standard applications 1200 rpm is possible. Table 4 gives a short overview of the installed motor power and the throughput rates for the processing of PET on different machine sizes.

Fig. 13: ZSK – Evolution of torque and volume
Table 4: Machine sizes and Throughput rates

<table>
<thead>
<tr>
<th>Machine Size</th>
<th>Power kW</th>
<th>max. speed 1/min</th>
<th>Throughput kg/h</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZSK 32McPLUS</td>
<td>20</td>
<td>400</td>
<td>30 - 80</td>
</tr>
<tr>
<td>ZSK 40McPLUS</td>
<td>42</td>
<td>400</td>
<td>50 - 160</td>
</tr>
<tr>
<td>ZSK 50McPLUS</td>
<td>82</td>
<td>400</td>
<td>100 - 325</td>
</tr>
<tr>
<td>ZSK 58McPLUS</td>
<td>126</td>
<td>400</td>
<td>150 - 500</td>
</tr>
<tr>
<td>ZSK 70McPLUS</td>
<td>226</td>
<td>400</td>
<td>300 - 900</td>
</tr>
<tr>
<td>ZSK 92McPLUS</td>
<td>502</td>
<td>400</td>
<td>600 - 2000</td>
</tr>
<tr>
<td>ZSK 119McPLUS</td>
<td>773</td>
<td>300</td>
<td>1000 - 3000</td>
</tr>
<tr>
<td>ZSK 133McPLUS</td>
<td>1131</td>
<td>300</td>
<td>1500 - 4500</td>
</tr>
</tbody>
</table>

8. Summary
The processing of undried PET is a proven technology. Above that there are significant savings with regards to energy and production costs in comparison with the single screw process. Coperion Werner & Pfleiderer has delivered more than 60 twin screw extruders with throughput rates of up to 3,000 kg/h for the PET production covering the whole range of films, fibers and bottle to bottle materials.