Conversion of PET-Bottle-Flakes to Added Value Products

Quality and Processing Criteria

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Ladies and Gentlemen,

Besides stagnation or even low growth rates in plastic production and processing, recycling of PET is a rather fast growing market segment. Depending on World region we observe annual growth rates between 8 and 25 %. Unexpected the US has reduced during the last year the PET recycling growth rate and it is one goal of our conference to inform about new technology and applications.

Besides the good mechanical and thermal properties of PET the quality of the final products is depending strongly on performance and process technology of collection, selection, cleaning and final processing.

Meanwhile the trend to circumvent the intermediate step of extrusion and chips cutting is ongoing and more and more nearly all conversion processes are using the purified bottle flakes as raw material.

My presentation today will provide you with an overview about the important products made of recycled PET and the essentials of flake quality and conversion technology. Target of development will be in any case to substitute step by step even virgin PET with the result of high profit margins.

CONTENT

1. The Flake Quality – Base of Success

→ Important impact to PET-flake quality is the collection system

→ Quality is decreasing in the row
Specific replacement of bottles out of refill systems – return vending
Bottles out of refund systems – deposit
Material collection systems – drop off
Household and industrial waste collection and separation – curbside

→ Contaminations
Colored PET green, blue, brown
Other polymers, PE, PP, PVC, MXD6, PVAC
Metals
Glue, seals
Syrup, flavor
Paper
Coarse environmental and foreign matter like sand, brick stone, glass fiber, fuel, mineral oil, house hold chemicals, agriculture chemicals

→ # Typical PET-flake specification

Typical PET Flake Specification – an Example

Type: Clear and colorless PET-Flakes, mechanical cleaned

Source: Water bottles, multi way or CSD, from deposit system or curbside waste collection (similar to DSD in Germany)

Viscosity (IV): 0,70 dl/g or 0,75 dl/g or 0,80 dl/g
→IV-spec. only possible from flakes from multi way selection
flakes out of deposit system or from curbside are mostly specified as > 0,70 dl/g

Levels of melt temperature: 245 - 250°C or 248 - 252°C or > 252°C
→Tm-spec. only possible from flakes from multi way selection
flakes out of deposit system or from curbside are mostly specified as Tm > 240°C

PET colored	< 10 000 ppm or < 1000 ppm or < 100 ppm
Polyolefin	<100 ppm or < 10 ppm or < 1 ppm
PVC	< 20 ppm or < 5 ppm or 0 ppm
Glue	< 20 ppm or < 5 ppm
Metal content	< 10 ppm or < 3 ppm or < 1 ppm
Water content	< 0,4 %
Color number "b"	< 5 or < 2
Bulk density	0,35 kg/dm³ or 0,45 kg/dm³

Example specification of bottle flakes: bottle grade

Property	Unit	Specification			Test Method
Color of Flakes		Clear 500MT	Green 200MT	Sky Blue 100MT	Per Month
Intrinsic Viscosity		0.72 - 0.76	0.72 - 0.76	0.72 - 0.76	
Particle Size	mm	Max 11	Max 11	Max 11	SYC - 6
Aluminum Content	ppm	Average 5	Average 5	Average 5	SYC - 4
PVC Content	ppm	Average 5	Average 5	Average 5	SYC - 25
Floatable	ppm	Average 5	Average 5	Average 5	SYC - 5
Moisture Content	wt%	Max 1.0	Max 1.0	Max 1.0	SYC - 8

Source : HANA Corp.

Example specification of bottle flakes: fiber grade

Property	Unit	Specification			Test Method
Color of Flakes		Clear 500MT	Green 200MT	Sky Blue 150MT	Per Month
Intrinsic Viscosity		0.72 - 0.76	0.72 - 0.76	0.72 - 0.76	
Particle Size	mm	Max 11	Max 11	Max 11	SYC - 6
Aluminum Content	ppm	Average 10	Average 20	Average 10	SYC - 4
PVC Content	ppm	Average 10	Average 20	Average 10	SYC - 25
Floatable	ppm	Average 10	Average 20	Average 10	SYC - 5
Moisture Content	wt%	Max 1.0	Max 1.0	Max 1.0	SYC

Source : HANA Corp.

Other Contaminations

For several critical applications like food bottles, clear A-PET film or spinning processes are the „invisible“ or finely distributed contaminations like

paper or cellulose fibers,
migrated chemicals or fuel,
destructed bottle surface areas and
precipitated water hardness CaO, MgO, Sulfates

of importance. To remove these contaminations the flake cleaning process must be more sophisticated.

→ Removing paper or cellulose fibers needs a final rinsing with fine filtered water.

→ Removing migrated chemicals or fuel could be supported by vented storage of flakes and is mainly performed by vacuum degassing during extrusion, and / or solid state polycondensation.

→ Removing destructed bottle surface areas needs a caustic treatment (NaOH).

→ Avoiding precipitated water hardness CaO, MgO, Sulfates needs the application of demineralized water during the last washing stage.

2. Flake Processing – Basics

Most of the processing technologies have the target to maintain or even increase the molecular weight (expressed as IV).

RESULT: An increasing number of processors like to avoid the conversion of PET-flakes to chips and instead of prefer the direct conversion to the final product.

Each melt process is bearing the risk of further downgrading by hydrolytic and thermal destruction.

→ Controlling or removing moisture is the most important process step.

Different ways to remove or control the moisture are

- → **State of the art crystallization at 150°C under agitation and drying at 150° – 180°C**
- → **Dry air drying under elevated temperature at 80°C without crystallization (dry air storage)**
- → **Vacuum degassing**
- → **Combination of pre-drying and degassing**
- → **Controlled drying or controlled moisture addition to reduce IV**

3. Product Outlets – Overview

During the last decade a steadily increasing number of products are made of bottle PET flakes. The very early started conversion of PET-scrap to staple fiber was later followed by increasingly higher performance products.

Product outlets

→ **Staple fiber, fiber fill, carpet fiber, non woven**
→ **Spun bond**
→ **A-PET film and sheet for thermoforming**
→ **Bottles for food and non food applications**
→ **Strapping**
→ **Monofilament**
→ **Technical yarn**
→ **Engineering plastic, polymer compounds,**
→ **Raw material for polyester producing processes**

Besides the direct conversion of bottle flakes to final products increasing amounts are recycled by partial glycolysis and addition of the glycolate to virgin PET production up to 25 %. Demand for fine filtration of glycolate.

Bottle Flake Processing Matrix

Product	Processing Steps			
	Flakes	Extrusion + Filtration + Chips	SSP	Final processes: Extrusion + Filtration +
Staple Fibers	X	(X)		Spinning
Spun bond	X			Spinning
A-PET film	X	(X)	(X)	Film casting
Bottles food	X (etching)	X + super clean	X	Preforming, bottle blowing
Bottles non food	X	(X)	(X)	Preforming, bottle blowing
Strapping, Monofil.	X	(X)	(X)	Spinning
Technical Yarn	X	(X)	X	Spinning
Engineering Plastic	X	X + compounding		Chips cutting
Raw Material for Polyester Production	X	(X)		Polycondensation

4. Important Products

4.1 Staple Fiber

A large quantity of the world wide collected PET bottles will be converted to staple fiber for different applications like textiles, fiber fill, carpet fiber and non woven. The processing is performed whether in smaller compact spinning units or via the re-granulation path in large extrusion spinning lines. There exists also the possibility to feed recycling melt as a side stream to continuously running direct staple fiber spinning plants.

Ways to produce staple fiber from PET-BOTTLE-FLAKES

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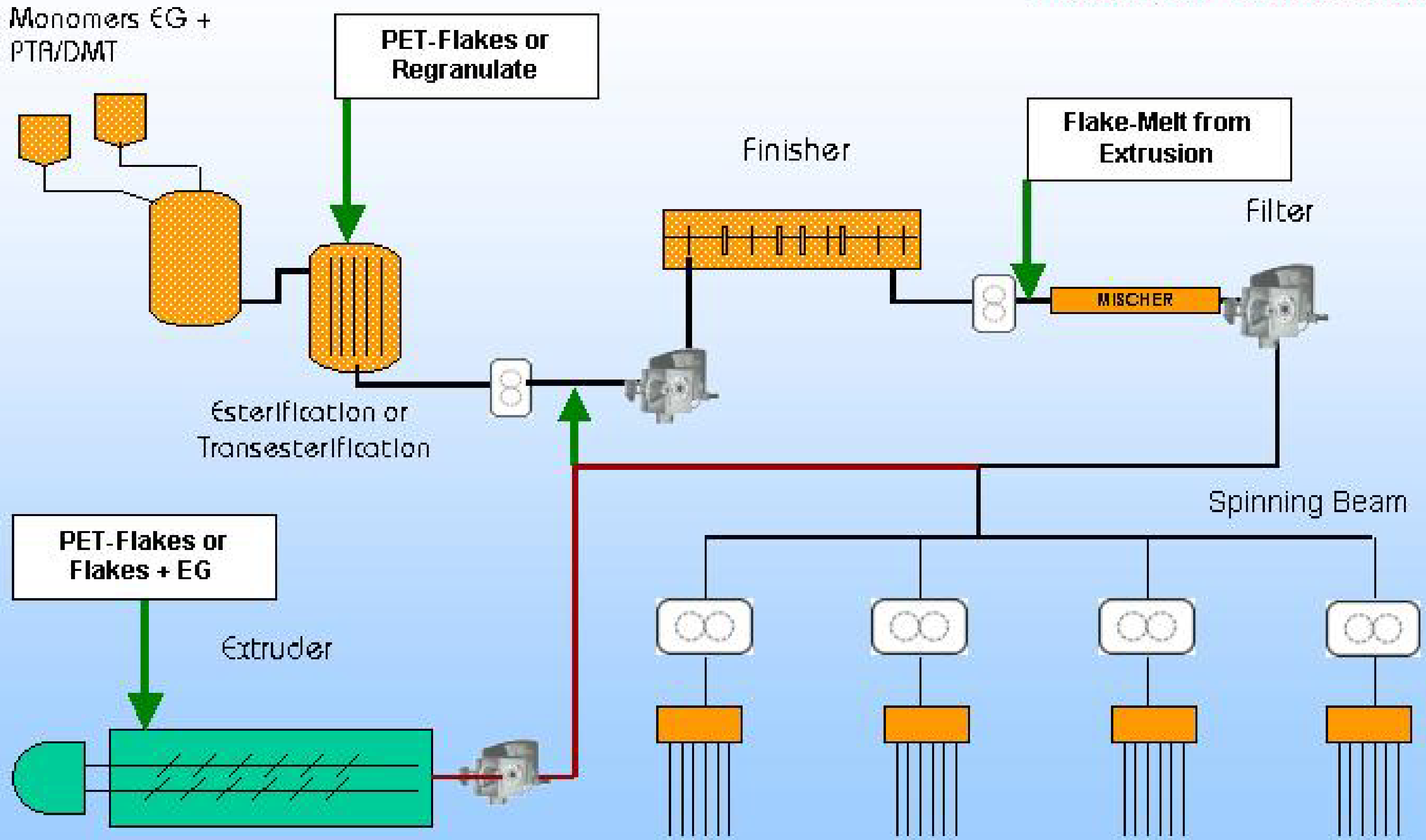


Staple fiber - IV adjustment:
Depending on collection source the flake IV can vary in a broader range from 0,68 – 0,80 dl/g. Scrap from CSD-bottles in tropical areas has the tendency to higher IV values.

Standard staple fiber IV is in a range of 0,58 – 0,62 dl/g. Depending on feed IV a certain IV-adjustment is desired.

One possibility is a controlled humidity process during extrusion to downgrade the PET-melt by hydrolysis. With fluctuating humidity content of the flakes this kind of process is difficult to control.
Second possibility is pre-drying to about < 100 ppm and addition of MW-modifier.

Melt filtration: To keep spin packs continuously and long time running a sufficient melt filtration < 60 µm is suggested. Sufficient equipment is the rotary filter RSF*genius.*

4.2 Bottle Chips - Food and Non Food Grade

→**Food grade:** IV-range is the same like virgin PET resin specification 0,72 – 0,85 dl/g, important difference to non food application is the implementation of so called super cleaning or super cycle process steps.

Different approaches in the market like OHL-process, BUHLER-process or SCHMALBACH-LUBEKA-process. All these processes containing intensive vacuum degassing during extrusion and solid state polycondensation. Meanwhile more attention is drawn to the flake treatment whereby the flakes are etched by diluted caustic soda.

Most important for the food grade recycling is the demonstration of FDA compliance of the whole recycling process technology by a special spiking of example toxics and the complete removal. Cost intensive are demonstration, extraction tests and chemical analysis.

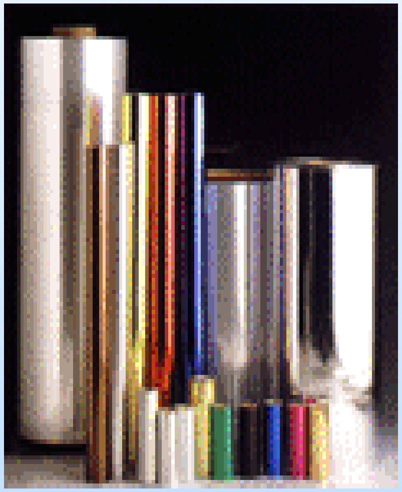
Melt filtration: A complete filtration in a range of 35 – 60 µm is necessary, no black spots are allowed. Continuous melt filtration is needed.

→**Non food grade:** Depending on bottle size and shape the resin IV can vary between 0,68 and 0,80 dl/g; for transparent bottles impurities are to remove by melt filtration, to provide comparable quality like virgin bottle resin the chips production contain the following steps:

Flake production and purification → extrusion, melt filtration 50 - 100 µm, chips cutting → crystallization and SSP

A very interesting area of packaging container is the fast growing section of paint and coating. Here PET is competing with metal cans as well as with other polymers.

4.3 A-PET Film



In case the A-PET film is used to produce packaging material with direct food contact like drinking cups for water, the same super cycle or super cleaning as used for bottle grade is necessary.

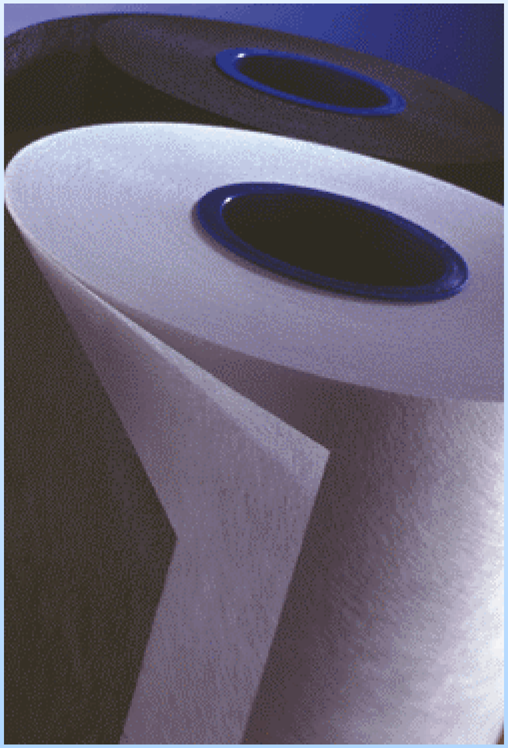
The IV-range of cast film is very broad and depends on the final conversion process and the desired mechanical properties. From recycled flakes without SSP film IV of about 0,65 – 0,70 dl/g is possible to produce. Higher IV is possible to reach with SSP.

For all other products many A-PET producers are changing with the steadily increasing flake quality from the film production process via the chips route to the direct conversion of PET-flakes to film.

Melt filtration: Similar to the bottle application a complete and continuous melt filtration is necessary because of the high visibility of defects, black spots and gels in the transparent cast film. Filter mesh 30 – 75 µm.

4.4 Spun Bond / Melt-blown

An interesting but know how demanding product is spun bond. Especially coarser spun bond articles like geo textiles, roof covering, reinforcement, filter or carpet back-materials are made of PET-flakes meanwhile.



The desired IV-range is more at the lower side between 0,58 and 0,68 dl/g which means that during processing some destruction might be applied in controlled manner.

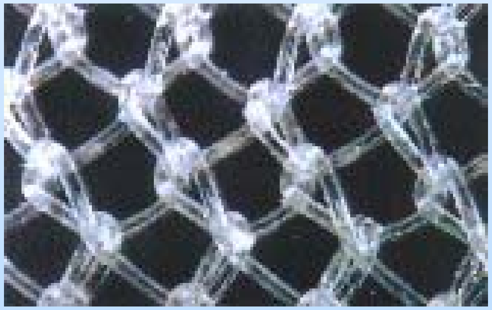
Direct conversion of flakes to spun bond is becoming increasingly of interest because of significant cost savings.

One special process to produce textile like products is the melt-blown process.

Melt filtration: As all spinning processes also spun bond spinning needs to have long spinning die operation time combined with a pressure constant melt in front of the melt metering pump. Desired filter fineness will be in a range of 25 – 60 µm, the recommended filtration system is RSF*genius.*

4.5 Strapping, Monofilament, High Tenacity Yarn

The conversion of bottle flakes to strapping, monofilament and technical yarns needs a higher or the highest IV in PET processing in the most cases. PET strapping for instance is of very high tenacity and can replace steel strapping. Monofilaments are widely used to produce gaze or wire-netting and technical yarns are covering a wide rang of applications. Excluded low tenacity and low quality products the polymer IV is in a range of 0,8 – 1,2, for some special applications even higher IV is desired.



Production process exists mostly of

→Extrusion including first melt filtration and chips cutting
→Crystallization and solid stating to lift up IV
→Extrusion, melt filtration, spinning, stretching, winding
→Meanwhile also direct conversion of bottle flakes is applied.

A wide variety of processing equipment is necessary

Melt filtration: Despite dimensions (diameter) of the final products are more at the larger and coarser side melt filtration is playing an important role. As higher the stretching ratio and as higher the stretching forces as more sensitive the stretching process depends on melt impurities which are week points.

Therefore it is recommended to filter during the low viscosity extrusion process as fine as possible. In this case a filter cascade existing of a coarse pre-filtration in a range of 100 – 200 µm and a second fine filtration in a range of 20 – 50 µm.

Fine filtration at the high IV process side is because of the higher delta-p more difficult

Reactive extrusion:

Slowly but stepwise reactive master batch to maintain or even increase IV are coming to the market. For several applications additives like STABAXOL- or PMDA- master batch are available.

4.6 Compounds like Engineering Plastics and Masterbatch

An increasing amount of bottle flakes are meanwhile converted to compounds, engineering plastic and masterbatch.

Especially filled and colored compounds and engineering plastic are possible to produce to a certain content based also on lower quality base flakes like colored flakes from curbside collection.

Masterbatch producer are using bottle flakes increasingly as cost effective matrix material. High flake quality and purity are requested (depending on final application).

Compared to the standardized staple fiber market, the engineering plastic sector is highly diversified with a large number of customized compounds and continuously changing quality demands which needs high amount of production know how and close customer relation.

Melt filtration: To maintain the mechanical properties and to avoid visible specs and spots a more coarse filtration in the range of 75 – 200 µm is recommend. Several technical solutions out of the Gneuss filter equipment are available.

4.7 Raw Material for Polyester Production

Bottle PET Resin

Especially because of the demand of Coca Cola and other environmental forces there is an increasing need to use recycled PET for the production of virgin PET-resin. This development is increasing the interest in PET-flakes as PET-raw material. For this application it is important to remove filterable and destructible impurities as early and as completely as possible. Especially impurities which are undergoing thermal destruction and discoloration must be removed.

Many of the currently in the market operating re-granulation facilities are working with too coarse and insufficient filter equipment.

PET-flakes are treated in many places unfortunately with processing methods like they are commonly used in the recycling of waste mass plastics like polyolefin or PS. Extremely high shearing and friction forces are applied which leads to mechanic-chemical destruction.

→ A large amount of heating energy (partly until the softening of the polyester) is created at agitation and cutting devices, which produces punctual extreme high peak temperatures with the result of discoloration

→ Therefore it is necessary to think about a general redesign of cutting, feeding and melting technology to preserve the high product quality of bottle bet best → twin screw extruder + melt pump + fine filtration

Besides PET-bottle-resin there are meanwhile numbers of polyester products which could be fed totally or partially by PET-bottle-flakes as raw material. The list beneath contains a collection of examples:

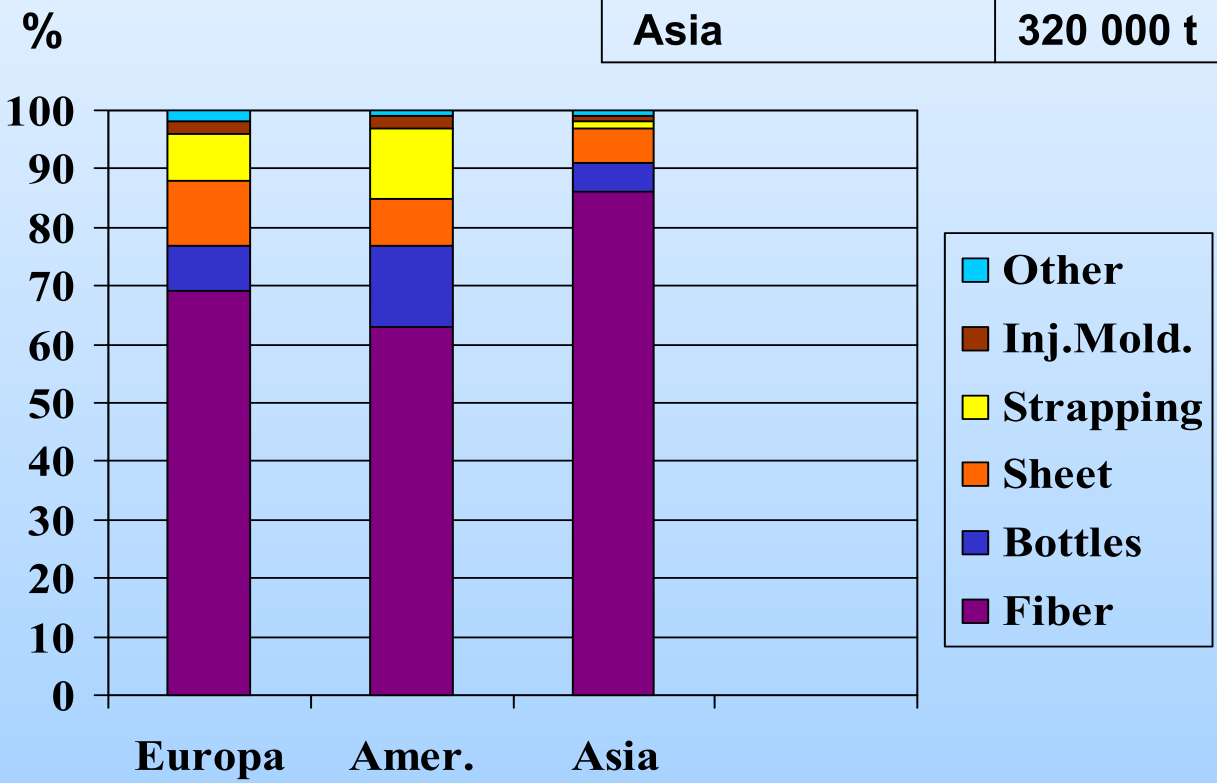
Textile polyester for staple fiber
Melt glue or hot melt
Low melt temperature polyester for spun bond
Powder coating
Melt coating
Base resin for paints and varnish
Biopolymers
Engineering plastics like PBT by glycol exchange

The quality demands for all such applications are:

→Easy to transport, feed and convey
→To process without additional equipment
→Neutral color
→Filtered at 20 – 50 µm depending on final application
→Consistent and reproducible composition and quality

Main application of bottle scrap

Collected Bottles	Year 2001
Europe	350 000 t
America	580 000 t
Asia	320 000 t



SOURCE: PCI

5. Summary

→ PET bottle collection and flake production are growing disproportionate to the bottle PET resin market.

→ Bottle flake quality improves steadily and is becoming increasingly standardized

→ The direct conversion of bottle flakes penetrates meanwhile nearly all polyester downstream processing technologies.

→ Most important product outlets are staple fiber, bottle resin for food and non food applications, A-PET cast film, spun bond, strapping, monofilaments and technical yarn.

→ Key to highly economical downstream processes based on direct conversion of PET bottle flakes is a high melt purity under pressure and process constant melt conditions.

→ The today's speakers are providing industrial matured solutions together with packages of engineering, know how support and experience in the field of PET recycling.

Often repeating abbreviations in the field of polyester

AA	Acetaldehyde
A-PET	Amorphous Polyester
BCF	Bulked Carpet Fiber
BD-1,4	Buthanediol-1,4
C-PET	Crystallized or Fast Crystallizing Polyester
COOH	COOH-End Groups of Polyester
Co-PET/PEN	Co-Polyester with 2 – 10 Mass % NDA
CHDM	Cyclohexanedimethanol
DEG	Diethylene Glycol
DMT	Dimethylterephthalate
DSC	Differential Scanning Calorimetry
EBM-PET	Polyester for Extrusion Blow Molding
EG	Ethylene Glycol
EO	Ethylene Oxid
FR-PET	Flameretardant Polyester
GC	Gas Chromarography
IPA	Isophthalic Acid
IV	Intrinsic Viscosity
LPG	Liquid Power Gas
MB	Masterbatch
Mn	Molecular Weight- Number Average
NaSiP	Sodium Sulphoisophthalic Acid
NDC	Dimethyl-2,6-naphthalenedicarboxylate
O2	Oxygen
OH	OH-End Groups of Polyester
PA6	Polycaprolactame
PBT	Polybutylene Terephthalate
PDO	Propylenediol or Propylenediol-1,3
PEG	Polyethlene Glycol
PEI	Polyethylene Isophthalate
PEN	Polyethylene Naphthalate
PETG	Glykol modified Polyester, stands for CHDM modified amorphous Co-Polyester
PLA	Polylactic Acid
POY	Partial Oriented Yarn
PTA	Purified Terephthalic Acid
PTT	Polytrimethylene Terephthalate
PX	Paraxylene
Sb	Antimony
SiO2	Siliziumdioxid
SSP	Solid State Polycondensation
TEG	Triethylenglykol
Tg	Glastransition Temperature
Tk	Temperature of Maximum Crystallization
Tm	Crystallite Melt Temperature
THF	Tetrahydrofurane
TiO2	Titandioxode
TMA	Thermomechanical Analysis
USG/UWG	Under Water (Strand) Granulator