PET BOTTLE TO BOTTLE RECYCLING

with the MRS Extrusion Concept

Dr. Axel Hannemann
Gneuss Kunststofftechnik GmbH

Technical Report No. 39

Contents

1. Introduction

The MRS technology is an attractive alternative to conventional recycling technologies and opens new possibilities with regards to flexibility, quality and energy efficiency.

The recycling of post consumer PET bottles to bottle grade chip is common practice today. There is however a clear trend toward ever increasing quality requirements in the future.

With the optimum technology, it is possible to recycle PET bottles almost indefinitely, thereby achieving a real recycling cycle.

One special characteristic of PET which can make its processing difficult is its hygroscopic behaviour. During storage and during the washing of post consumer bottles, water molecules from airborne moisture or from surface contact diffuse into the PET. During the extrusion process, these water molecules break down the molecule chains (hydrolysis) resulting in a drastic reduction of the viscosity, unless the water is removed.

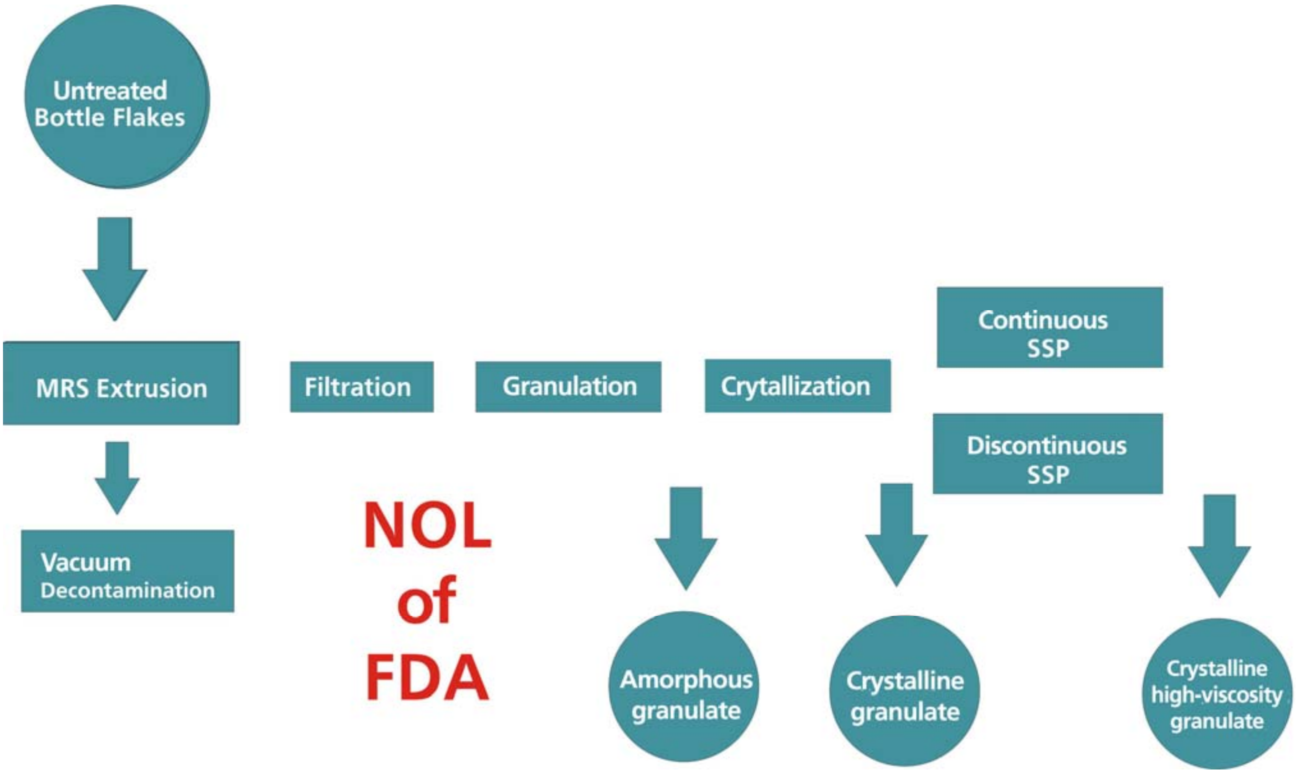
Manufacturing Process:

After collection, coarse separation of foreign particles and materials takes place. Subsequently, the bottles are ground into flake. These flakes are washed in a hot wash process where dirt, labels and adhesive from the labels is removed. In a floatation bath, polymers are separated according to their specific gravity. The most common foreign polymer, PP (bottle caps) are floated off. The PET bottle flakes are then placed in a centrifuge to remove the surface water. Metals are separated out and further separation of PVC and in most cases, colour sorting takes place.

This bottle flake is then processed on an extruder. The PET is melted and homogenised and filtered. The filtration system uses a woven wire mesh filter element to collect typical contaminants such as sand and other fine particles.

To ensure a homogeneous melt, a static mixer is typically used.

The gentle treatment of the polymer in the MRS extruder and the fact that the material processed on this extruder means only relatively short residence times in the SSP result in a low amount of yellowness and excellent transparency.



Subsequently, the PET is pelletised. Depending on the type of pelletising process used, the result is spherical or cylindrical PET pellets or chips.

These pellets or chips are dried, crystallised and go through an SSP process (solid state polymerisation) where the viscosity is raised to the required level.

Depending on the throughput rate, this final processing stage can be a continuous process (for larger throughput rates). For smaller throughput rates, typically the more flexible discontinuous processes are used.

Thanks to its great flexibility, the MRS concept is particularly well suited to the processing of smaller flows of materials of different types (colour, source). If combined with a discontinuous SSP whereby the viscosity can be set individually for each batch.

The PET chips or pellets can be used for the most diverse applications: for fibres, sheet for thermoforming and of course for injection moulding preforms for bottles.

Additionally, it is possible to produce chips or pellets from up to 100 % post consumer bottles for products which come into direct contact with foodstuffs.

The MRS extrusion process has a LNO (letter of non objection) from the FDA for processing 100 % PET bottle flake to food contact products both with and without a subsequent downstream SSP process.

DEPARTMENT OF HEALTH & HUMAN SERVICES — Public Health Service

Food and Drug Administration
5100 Paint Branch Parkway
College Park, MD 20740

January 26, 2011

Frank Welle
Fraunhofer Institut
Verfahrenstechnik und Verpackung
Giggenhauser Str. 35
D-85354 Freising
GERMANY

Re: Prenotification Consultation (PNC) 991

Dear Mr. Welle:

This letter is in response to your electronic submission, received on October 20, 2010 (PNC 991), requesting on behalf of Gneuss Kunststofftechnik GmbH (Gneuss) a non objection letter from FDA confirming the capability of Gneuss's secondary recycling process (a so-called "Super Clean" process) to produce post-consumer recycled polyethylene terephthalate (PCR-PET) pellets that are suitable for use at levels up to 100% recycled content in the manufacture of PET containers for contact with all food types under Conditions of Use C through G, as described in Table 2, which can be accessed from the Internet in the Ingredients and Packaging section under the Food topic of www.fda.gov.

We have reviewed Gneuss's recycling process as well as the information obtained from surrogate testing and migration modeling which were submitted to demonstrate the capability of the Gneuss's secondary recycling process to remove potential contaminants from PCR-PET. Based on our review of these data, we have determined that Gneuss's secondary recycling process, as described in the subject submission, would be effective in reducing potential contaminants from PCR-PET to levels that result in dietary concentrations not to exceed 0.5 ppb, FDA's threshold of regulatory concern. This determination covers the use of PCR-PET derived from the feedstock that consists of post-consumer food and non-food PET containers (excluding industrial PET containers), and the PCR-PET complies with the existing applicable authorizations.

We have concluded that Gneuss's secondary recycling process, as described in the subject submission, would produce PCR-PET pellets that are suitable for use at levels up to 100% recycled content in the manufacture of articles for contact with all food types under Conditions of Use C through G, as described in Table 2, which can be accessed from the Internet in the Ingredients and Packaging section under the Food topic of www.fda.gov. If Gneuss's recycling process is modified, new data may need to be evaluated.

Fig.1: Letter of Non Objection from FDA

2. Processing of PET

2.1 Drying (moisture extraction) and extrusion

2.1.1 The importance of moisture extraction in the PET extrusion process

Today, the established technology for the minimisation of degradation of the PET is to dry the material before processing it on a single screw extruder. The drying of PET is complicated, maintenance intensive and expensive in terms of investment costs, factory space and running costs due to the high energy consumption as the material is typically dried using dry air at 180°C. The fact that the material has to be dried for up to 8 hours also reduces the PET processor's flexibility.

In the processing of PET with an extruder at normal processing temperatures, a chemical process takes place in that any water molecules which may be present reduce the PET chain lengths which results in a reduction in the viscosity which in turn reduces the mechanical properties of the final product.

In order to avoid this problem, the pellets are conventionally pre-dried over a long time period in order to remove the moisture prior to extrusion in order to prevent hydrolysis.

This chemical reaction is however ***reversible*** and the point of equilibrium can be driven to one or the other side by reducing or increasing the water content.

This phenomenon can be used to positive effect during extrusion. By removing the water molecules during processing, the damaged PET molecule chains can rebuild themselves.

The physical prerequisites for the removal of water molecules are described by the diffusion process, which can be formulated with the help of the Fick´s first law:

$$\frac{\Delta n}{\Delta t} = - D \cdot F \frac{dc}{dx}$$

This law shows that the quantitiy of water removed (Δn) will increase in relation to the residence time (Δt) the larger the melt surface area (F) and the driving concentration incline (***d***c) and the thinner the polymer layer (***d***x). In other words, the better the polymer is mixed and the better the surface is exchanged, the more the chemical equilibrium will move toward long chain molecules with better mechanical properties

2.1.2 Devolatilisation with the MRS extrusion concept

With the MRS extruder, PET pellets are processed direct, without pre-drying the material. During extrusion (in the melt phase) the polymer surface is greatly enlarged and exchanged at an extremely high rate, thereby enabling volatiles to be extracted. (Fick´s law of diffusion).

The MRS (Multi Rotation System) extruder can be described in general as a single screw extruder with a very special degassing section. The polymer melt is delivered into a large single screw drum.

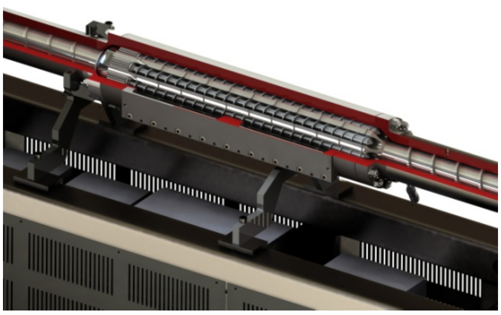


Fig. 2: Multi Rotation Section

The drum contains 8 or 10 (depending on the model size) small extruder barrels, parallel to the main screw axis.

Installed in these small extruder barrels are the "satellite" screws, which are driven by a ring gear in the main barrel. The satellite screws rotate in the opposite direction to the main screw while they rotate around the screw axis. This disproportionately increases the surface exchange of the polymer melt.

Fig.3: Transition of Multi Rotation Section to Discharge Section

The extruder barrels which are cut into the drum of the multi rotation system are approximately 30% open to ensure the optimum melt transfer into the barrels and so that the evacuation can take place without restrictions. Further, precise control of the melt temperature is possible as the temperatures of all the surfaces in contact with the melt can be controlled accurately.

Fig.4: MRS Extruder

Thanks to its patented multiple screw section, the MRS makes a very large area available and permits unmatched degassing performance, even with a vacuum of only 20 to 40 mbar.

This makes the processing of undried flakes or pellets with up to 1 % residual moisture (10.000 ppm) possible.

This evacuation technology is based on the robust and proven single screw extruder concept. In the Multiple Rotation Section, there are 8 satellite screws oriented in a ring pattern in the main MRS drum.

In this way, the MRS avoids the problems of alternative multiple shaft or screw designs (intermeshing) which are considerably more sensitive to mechanical damage due to their tight clearances. This last point can be decisive in the reprocessing of PET bottle flake which frequently includes coarse contamination.

Further positive effects of the MRS technology are the 100% dehumidification of PET as well as the possibility of increasing the intrinsic viscosity of this material.

Thanks to the multi rotation elements, a melt surface is made available which is far greater than that of conventional extruders. For example: the MRS system creates a melt surface exchange rate which is 25 times greater compared with a co-rotating twin screw extruder.

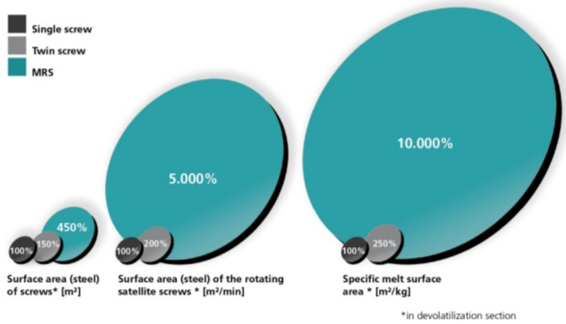


Fig.5: Comparison of different extrusion concepts

The melt treatment is extremely gentle, resulting in a particularly high quality final product for example with regard to yellowness.

Compared with other multi screw systems, the MRS is characterized by its extremely compact and rugged design. The rotating satellite screws run in individual bearings and are therefore comparable with a drum containing a number of single screws.

The evacuation or degassing system is modular and can be ideally matched to the individual requirements, thanks to its remarkable performance. The position, length and design of the modules can be varied.

A South American customer manufactures sheet for thermoforming from 100 % post consumer bottle flakes. These are processed without any pre-drying even though the flakes can have a moisture level of up to 10.000 ppm. The I.V. of the material was analysed at various stages along the extruder, upstream and downstream of the devolatilising section, the I.V. of the final product was likewise analysed.

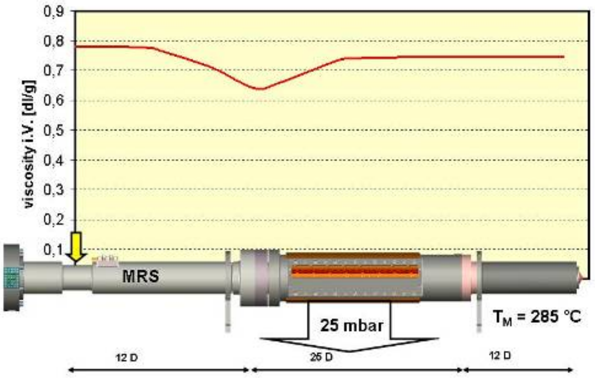


Fig.6: Viscosity when processing PET –Bottle Flakes

It is obvious that in the very first section of the extruder the viscosity of the polymer is reduced, because the water content is integrated into the chains of the PET. The more moisture is in the input, the more the decrease. In the degassing section these water molecules are extracted by vacuum (driven by diffusion), here the reversed chemical reaction takes place. Therefore the MRS is able to increase the chain length, the molecular weight, the viscosity and the mechanical properties of the polymer. The viscosity built up can be controlled by the vacuum level itself; consequently it is possible to use an online viscosity measurement to control the vacuum in order to stabilize the viscosity level by a closed control loop.

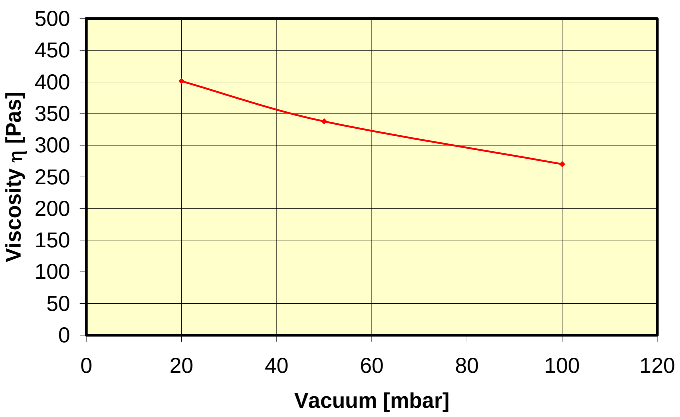


Fig.7: Correlation between viscosity and vacuum applied

The viscosity increase in the devolatilisation section is influenced and controlled by the vacuum level. The influence of the vacuum on the viscosity is considerable.

The increase in viscosity in response to the increasing vacuum level can be seen clearly.

2.1.3 Advantages of the MRS technology for Bottle to Bottle Recycling

The MRS Extrusion system offers the highest level of flexibility. This extruder can be fed with both crystalline and amorphous pellets / flakes with varying viscosities. The viscosity of the material leaving the extruder can be adjusted individually via the vacuum degassing system to suit the requirements of the application.

Amorphous pellets or chips made from PET processed on the MRS with a defined viscosity can be processed immediately. Alternatively, the pellets or chips can be crystallised and can even be processed in an SSP where higher viscosities are required.

2.2 Online measurement and control of the processing parameters

Today, the monitoring, logging and analysis of processing parameters is a key element in plastics processing in order for example to be able to trace problems relating to a customer complain but also to be able to react as quickly as possible to changes in these parameters. Through this monitoring, a continuously high product quality can be ensured.

During extrusion, the most important factors with regard to the properties of the final product are the melt temperature, the melt viscosity and the melt pressure(s) in the machine. With the help of these parameters, the failure of heater zones or cooling fans, variations in the raw material parameters and wear in the line can be monitored. Dangerous situations can be avoided if equipment is for example automatically shut down if a too high pressure increase takes place quickly.

Through the use of melt pressure and melt temperature sensors and with a known throughput rate and known geometry, the flow resistance of a liquid can be calculated and from this, the dynamic viscosity of the medium can be extrapolated. This is a measurement of the average molecular weight of the polymer which defines its properties such as elasticity and stretch or elongation.

2.2.1 Process control with the Gneuss Online Viscometer VIS

A small part of polymer melt is diverted from the main melt channel and with a high precision gear pump it is pumped through a precisely manufactured slot capillary. Melt temperature and melt pressure (at two locations) is measured. Based on internal calculations the viscometer monitors the value of the representative shear rate and the corresponding viscosity.

The viscometer can be fitted between two flange connections. The melt channel can be designed according to customers' specifications between 0.5 and 2 mm. The unit includes a pump drive, a pump, pressure transducers, temperature sensors and the control and evaluation electronics. The setting of process parameters, the evaluation and the display is realized via a user friendly touch screen panel or alternatively can be integrated into an existing control system.

Thanks to the Online-Viscometers VIS it is now possible to maintain the melt quality within a very narrow bandwidth in spite of varying input material conditions (residual moisture). The viscosity, measured by means of melt pressure and temperature is used as a control value to automatically adjust the vacuum in the devolatilising section of the MRS extruder, thereby guaranteeing constant melt properties (viscosity, molecular weight and therefore the mechanical properties which result from these), see fig.8.

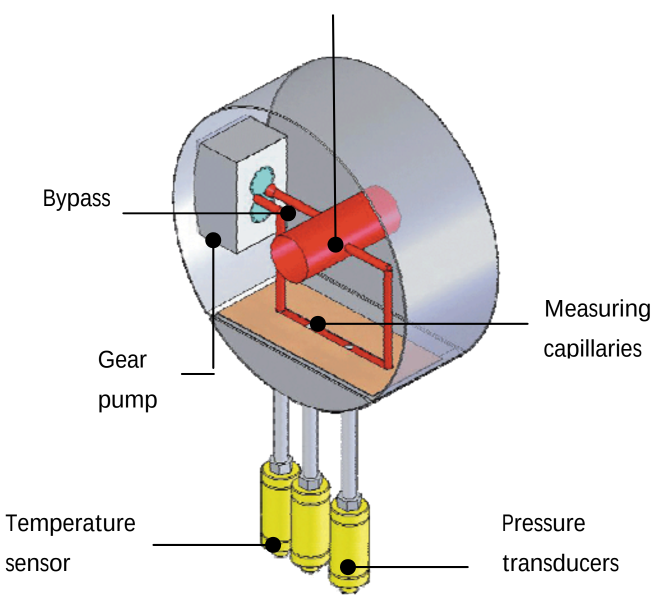


Fig.8: Online Viscometer VIS –technical layout

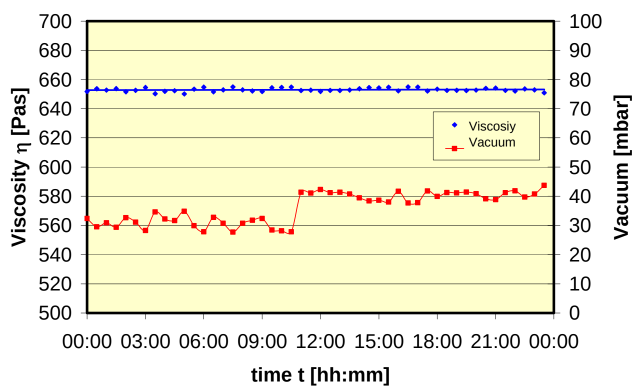


Fig.9: Viscosity in relation to residual moisture variations

The above graph shows the behaviour of the vacuum (red) and the melt viscosity (blue) over time. The change of input material at 11:00h can clearly be seen. The material silo was changed, but prior to this, the material was drawn from the lower section of the silo, this material having relatively high residual moisture content. Immediately after the change, the residual moisture level of the flakes is considerably lower. By increasing the vacuum in the devolatilising section of the MRS extruder, the viscosity and therefore the molecular weight of the material can be maintained, consequently maintaining the mechanical properties i.e. the quality of the final product constant.

2.3 Not only for recycling: melt filtration as a key element with regard to quality assurance and the economic aspects of the process

Before the polymer enters the die, all kinds of contamination should be removed, in order to achieve the highest quality requirements with regard to transparency (sheet) or tensile strength (sheet for thermoforming, strapping tape) and of course to protect the downstream components from damage. The Rotary Melt Filtration Systems from Gneuss are particularly suited to this purpose as they operate fully automatically and pressure constant.

*2.3.1 The RSF**genius**: process and pressure constant melt filtration with integrated self-cleaning*

The RSF***genius*** consists of three main parts – an inlet block, an outlet block and a filter disk rotating between them. The system is sealed by a metal to metal sealing with very narrow gaps as well as very hard and flat surfaces. It is guaranteed that all components in contact with melt are not in contact with the environment (e. g. oxygen).

The screen elements are located in a ring pattern on the filter disk, moving through the melt channel. When melt flows through the screen, hard particles get caught and the differential pressure increases slightly. The control system reacts to this pressure increase and makes the filter disk index by 1-2 angular degrees. Thus, contaminated screen area is continuously moved out of the melt channel and clean screen area is moved into the melt channel without changing the active filter area. Due to this mode of operation, the filtration system operates process- and pressure-constantly. The variation of the pressure differential across the filter (Δp) amounts to max. 2 bar.

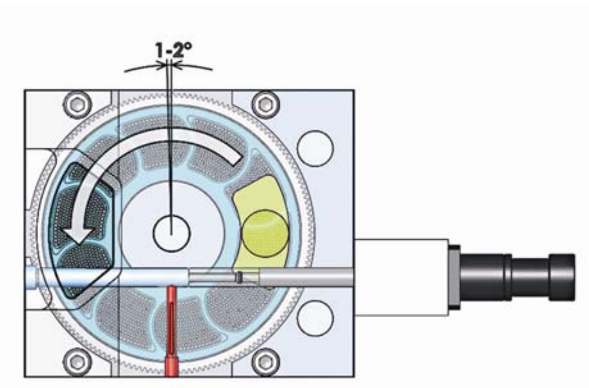


Fig. 10: RSF***genius*** - Technical Layout

Cleaning of the contaminated screens takes place just before they are re-introduced into the melt channel. The dirt cake is then cleaned via a high-pressure segment purging system. For this purpose, already filtered melt is taken from the outlet block and siphoned into a hydraulically-driven piston and then shot from the back at high pressure, through the filter disk and through the screen into the inlet block. The purging pressure is measured and can be adjusted and optimized. Only a small segment (approx. 1% of the screen area) is cleaned at a time with a defined high impulse. The stroke of the back-flush piston (= melt amount to be used) as well as the speed (= intensity of cleaning) can be adjusted freely and therefore optimized. So on one hand the cleaning is very efficient but on the other hand the melt amount used is minimized (backflush losses are minimal).

Due to this mode of operation the screens can practically be cleaned 100% and can be re-used, depending on the filtration fineness, up to 400 times. This makes a fully-automatic filtration (without any operator attention) possible for several months in some applications.

Thanks to the RSF***genius***, even heavily contaminated polymers can be reprocessed into a high value nonwoven.

3. Case example application

A manufacturer of PET flakes chose to extend the value addition of his production by installing an extrusion line with a downstream SSP.

With the MRS Extrusion concept, this company was able to avoid cost intensive, complicated and potentially damaging pre-treatment of the flakes prior to extrusion.

PET is processed with a throughput rate of up to 2.000 kg/h from washed bottle flake to a high value PET bottle grade chip / pellet.

The MRS extruder, operating at a vacuum of only 25 to 30 mbar permits very short residence times in the downstream SSP. The Melt Filtration System type RSF***genius*** filters the material from 75 µm to as fine as 30 µm.

The Online Viscometer measures the viscosity of the polymer upstream of the die and is equipped with a control loop to regulate the vacuum on the MRS extruder, thereby ensuring that the viscosity is automatically held at a controlled, constant level.

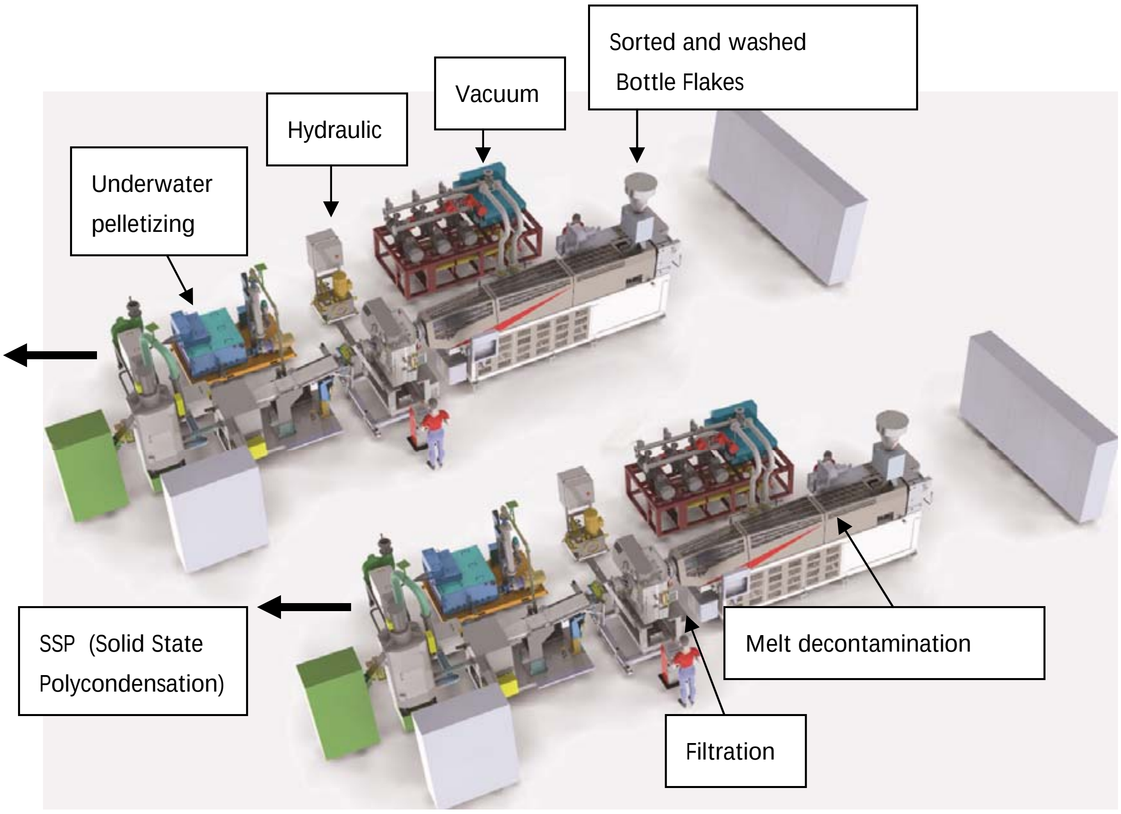


Fig.11: Arrangement drawing of extrusion equipment

Compared to conventional single screw processing with crystallisation and pre- drying down to a water content of less than 50 ppm the specific energy consumption is much less, around 20%, because of the immense energy costs for heating the pellets by hot air during the preparation for extrusion.

In comparison to twin screw processing of the flakes the energy consumption is also less, because a high vacuum of less than 5 mbar is needed with the twin screw and in addition to that normally the flakes have to be pre-dried.

The specific energy consumption of the MRS technology is 15% to 25 % less than conventional technologies, not to mention the higher flexibility and less maintenance on dryers and high vacuum systems.

	MRS Wh/kg	Twin Screw Wh/kg	Single Screw Wh/kg
Cristallisation	-	-	90 *
drying < 50 ppm	-	-	120 *
Pre – drying to 1000 ppm	-	60 *	-
extruder drive and heating	295	230 *	240 *
vacuum	45	90 *	-
Booster pump	-	30 *	-
total	350	410 *	450 *

* Specifications given by our customers

Table 1: PET processing: comparison of energy consumption between extruder types

4. Conclusion

The revolutionary new MRS Extrusion System ensures extremely efficient degassing of the polymer melt due to its unmatched polymer surface area exchange rate and by the way that the exchange takes place.

Consequently, it is for example possible to process PET bottle flakes without drying to bottle grade pellets.

The MRS Extrusion System (including the Multi Rotation Section) is based on the rugged and simple single screw extruder and is therefore perfectly suited to the processing of highly contaminated polymers such as post consumer PET bottles.

Thanks to the FDA Letter of Non Objection, the MRS Extruder, its Devolatilisation System and the Rotary Melt Filtration System permit the processing of up to 100 % unwashed PET bottle flakes to containers for food and beverage contact.

Since the MRS extrusion system requires the input material to be neither pre-dried nor crystallised, the system is an economically attractive alternative to the conventional technologies for processing PET as the factory floor footprint can be reduced by 25 % and the energy requirement by 25 %.

The MRS has already been proven as a reliable and practically maintenance – free system for several years on a range of different applications.

Imprint

No. 39/11
Dr. Axel Hannemann
Moenichhusen 42
32549 Bad Oeynhausen
Germany
Phone: +49 5731 / 5307-0
Fax: +49 5731 / 5307-77
Email: gneuss@gneuss.com
www.gneuss.com