Is a ready-made polyester polycondensation pilot line possible?

Combination of standard equipment as solution

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Content

1. State of the art pilot polycondensation in polyester development

Common continuous pilot facilities to produce linear polyester like PET or PBT are including all steps of the PET synthesis as they are

paste preparation,
esterification with water separation column
pre-polycondensation under vacuum ~ 100 mbar
polycondensation under 1 mbar
pelletizing.

The equipment is in general the same like in common continuous PET plants used. The capacity of such pilot lines is in a range of 50 – 200 kg/h. These are high expenditures on equipment and not to forget the labor facilities for preparation and analysis of raw materials, liquid/solid intermediates and polymer products.

To run such a pilot line needs qualified laboratory and plant staff available for three shift operation. All together there are immense financial and personnel efforts required.

To reduce costs for general polyester development the experimental work is mainly executed in discontinuous operating multifunctional autoclaves where esterification and polycondensation are executed step by step.

This advantage is most of the time opposed by low reproducibility of polymer quality and limited sample/product amounts. Some companies are using also their decommissioned small discontinuous productions lines for their development work.

2. Current R&D situation in the polyester industry

-Standard polyester technology for textile and packaging are matured

Different to the years 1980 – 2010 where the production process of polyester for textile and packaging applications was fast growing and highly dynamic we have the situation today that technology

development regarding process technology, plant capacity and equipment reached a saturation plateau. World scale plants are producing 800 – 1500 t/d in melt phase and solid phase. Main target today is reduction of conversion cost combined to clever product distribution and site logistics.

-Most polyester companies are producer only today

As a consequence of the above said most of the polyester plants today are producer only. When we are looking back to the last 5 to 10 years we saw for instance the tremendous growth of Indorama. They bought lots of partially inefficient PET plants round the World. Cost cutting is the tool making those plants efficient.

-Ongoing R&D significantly reduced

Within this development no further basic R&D is required and necessary. The logic consequences are reducing any kind of overheads and with this closing of laboratory and pilot lines. Even large polyester producer like INVISTA mothballed their continuous pilot facility recently.

-Specialties like PETG or CD-PET are produced in large scale

Another reason why polyester pilot facilities disappear is the fact that most of well-known polyester specialties like PETG, cationic dye able polyester, film-PET or a broad variety of bottle-PET are produced on large scale continuous plants.

-R&D concentrated on special polymer and bio polymer

Ongoing R&D is observed for special co-polyester, bio-polymers like PLA, bio-based PET or PEF. This is compared to textile and bottle PET a vanishingly small sector.

3. Polyester Recycling

The polyester recycling industry which is presenting meanwhile a world production capacity of about 10 Mio t/a is growing faster than virgin polyester production.
Reasons are at the one hand the annual growth of virgin polyester and at the other hand the additional growth by increasing environmental efforts to reduce raw material consumption in general.
Major products out of all the collected polyester waste are still all kind of textile intermediates like staple fiber and filaments. But also carpet fiber and technical fiber, strapping, cast film and bottles are meanwhile made fully or partially from recycled polyester.
It is to observe that a growing number of recyclers are active in up-integration means flake and regenerate producers are entering in the production of polyester intermediates or even final products like knitting, weaving, strapping, carpets or injection molded parts as examples.
Especially this industry is interested to modify, adapt or develop tailor made r-polyesters which satisfy the prerequisites of their final product quality.
Till now there is no equipment in the market which fits to the need of the polyester recycling industry to take substantial polymer product development in their own hands which is

- simple in design
- easy to operate
- fitting to the polymer processing environment
- usable as pilot and as small production line at the same time
- affordable in its investment and operation

4. A polyester pilot line off-the-peg?

The common approach to execute substantial development work in polyester is too expensive as learned under item 2. This means one has to simplify all process steps and select equipment which is available in the market.

-Major prerequisite is process and handling simplification

Looking at the polyester production process the steps paste preparation, esterification and pre-polycondensation are the most complicated one, whereas paste dosing, rectification during esterification, liquid handling and reaction water removal are demanding a fully equipped chemistry plant.
All those operations are fare away from experience and education in mechanical polymer recycling like sorting, washing and re-pelletizing.
To radically simplify the PET production process one could skip esterification and pre-polycondensation and use as start product standard polyester pellets or bottle flakes without additives of low or medium molecular weight.
Ideal market products in this quality are transparent and bright PET bottle flakes, film polyester pellets, bright textile polyester pellets or bottle PET pre-curser within an IV-range of 0,55 – 0,65 dl/g

-Some polymer chemistry

Polyethylene terephthalate is a living polymer. Under reaction temperature > 260°C the polymer melt reacts with diols under glycolysis and with dicarboxylic acids under acidolysis.
After sufficient reaction time and melt homogenization diols or di-acids are molecular disperse distributed. The present amounts of catalyst like Sb or Ti are efficient to support this reaction.
After successful reaction by acidolysis or glycolysis a subsequent polycondensation under vacuum is required to achieve the targeted molecular weight.

- Polymer modifications as example

Acidolysis: Addition of carbondiacids like isophthalic acid (IPA), sulfoisophthalic acid(SIPA), succinic acid (SA) , adipic acid (AA) or naphthaline dicarbonic acid (NDA)
Glycolysis: Addition of diols like diethylene glycol (DEG), triethylene glycol (TEG), polyglycole (PG), butanediol-1,4 or cyclohexanedimthanol (CHDM)
Modifier examples: pyromellitic anhydrite, pentaerythrite
Additives: stabilizer, toner, AA-scavenger, filler

Low melting and slow crystallizing polyester containing up to 5% IPA, NDA or Succinic acid (diol balance by addition equimolar amounts of diol)
Low melting polyester containing up to 5% diol like DEG, TEG , CHDM
Fast crystallizing polyester by exchanging MEG by BD-1,4
Polyester with special additives or chain branching agents

5. Smart combination of MRS and JUMP from Gneuss Kunststofftechnik GmbH (www.gneuss.com)

- The equipment

After five years of development Gneuss Kunststofftechnik GmbH is now ready to offer all pilot facility parts out of one hand. The two main equipment parts are the MRS and the JUMP-reactor.
The MRS allows processing all raw materials like virgin PET pellets or PET bottle flakes without any pretreatment like crystallization or drying.
The vacuum unit of the MRS which is able to run at medium vacuum in a range of 100 – 25 mbar or at high vacuum in a range of 5 – 0,5 mbar guarantee efficient removal of water which is causing IV-losses.
When PET-bottle flakes are used the different vacuum systems are able to remove present bottle additives which might disorder the polycondensation process or cause discoloration.
The JUMP is a compact polycondensation unit with proprietary internals which allows remarkable polycondensation progress within a moderate reaction time ~ 30 min.
As example: Standard polyester input material like transparent and colorless bottle flakes, IV 0,78 dl/g are converted by MRS under 20 mbar vacuum to PET-melt of IV 0,68 dl/g. The subsequent polycondensation under vacuum ~ 1 mbar is performed in the JUMP reactor. The IV of the pellets is adjustable within a range of 0,75 – 0,85 dl/g.

The feasible capacity of such kind of pilot installation is within a range of 50 -2000 kg/h. Those 50 – 2000kg/h pilot lines are also useful to produce larger polymer quantities for market introduction ad even for regular specialty production

With the ongoing product standardization on the one hand and the capacity growth of regular bulk production on the other hand small, simple and high flexible production units which are able to produce tailor made polyester products are not only a challenge for the polyester recycling industry but even a new chance for regular PET producer to simplify product development or produce special niche products.

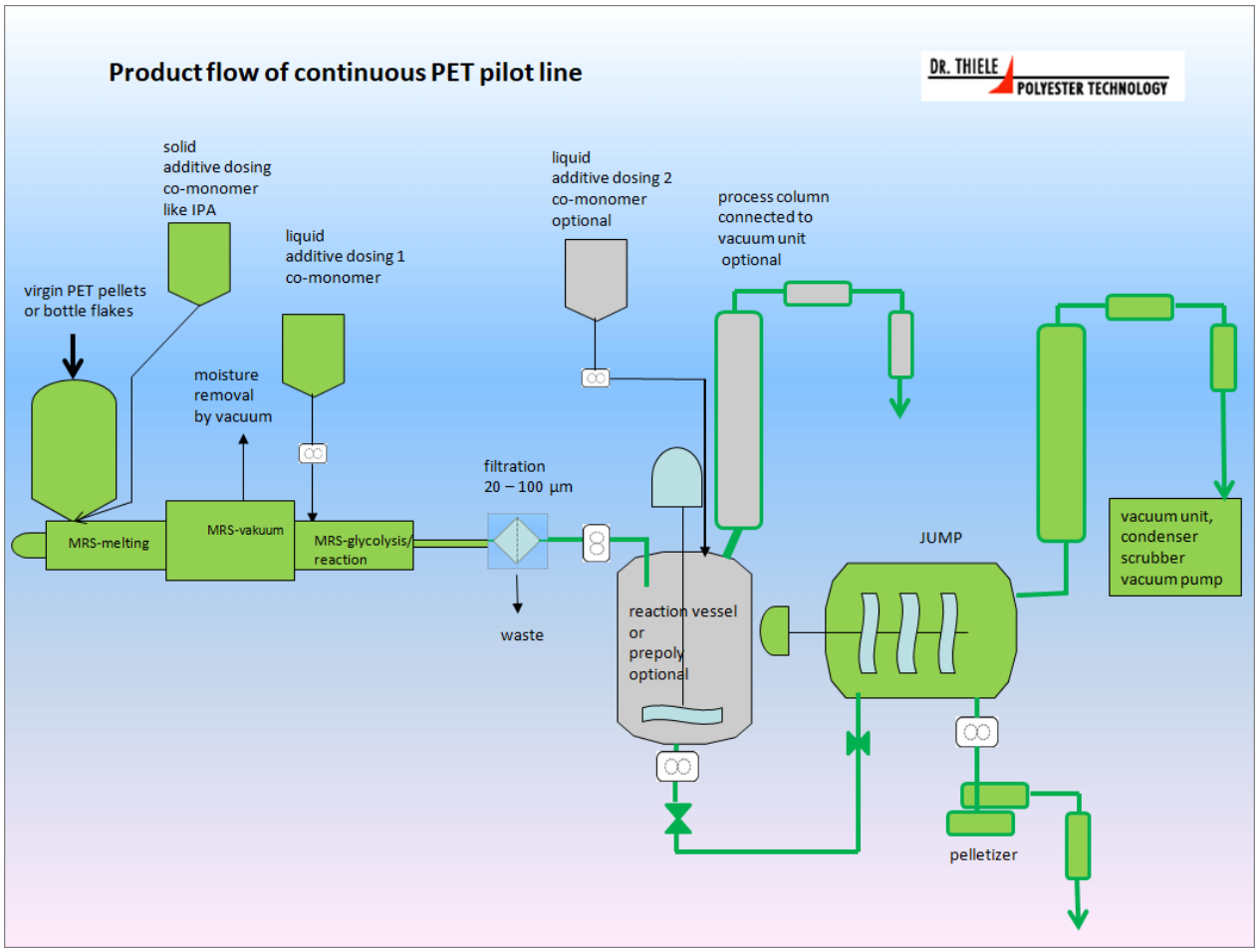
- Pilot line sketch

Figure 1 “Product flow of continuous pilot line” is showing the base configuration existing of MRS + JUMP. This layout allows producing polyester modifications with different additives and limited co-monomer introduction. As soon as substantial recipe changes are desired and co-monomers in a range up to 10% are added an additional residence time vessel (greyish colored in Figure 1) is required.

This residence time reactor guarantees the molecular disperse distribution of the additives or co-monomers. Residence time in this reactor is up to 60 min.

Depending on the required flexibility this reactor can be equipped with additional dosing units, backflow column and connection to the vacuum unit.

Figure 1 “Product flow of continuous pilot line”



6. Summary

Commonly used and state of the art pilot facilities to execute substantial polymer development in the field of linear polyester or gradual polymer modification are

- expensive
- complicated
- deeply involved in chemistry and analysis
- step by step disappearing
- uneconomical

The polyester industry is matured and has changed to large capacity bulk production units. Development is mainly focused on capacity growth and cost saving at the expense of flexibility.

Polyester recycling industry is still vitally growing. Targets are to increase the product range and profitability. The material input properties mainly from fiber and bottle waste are narrow limited. A broader range of polymer adaptation and modification is required to generate products of added value.

Suggested solution is polyester modification and material development by a simplified process existing of base polymer modification by acidolysis, glycolysis and subsequent re-polycondensation.

Combination of common equipment and process simplification are offered as pilot facility by the newly developed configuration existing of Gneuss MRS vacuum extrusion and JUMP polycondensation with the option of reaction / residence time extension by adding a simple stirring autoclave.

The new pilot equipment is usable for development, trial production and market introduction as well.