

GENERAL RECOMMENDATIONS FOR EXTRUSION

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1. EXTRUDER

1.1. EXTRUDER TYPE

- single screw extruder
- polyolefin type

1.2. SCREW - BARREL

- polished barrel (or very small grooves)
- barrel - screw tolerance : 200 microns
- screw geometry :

PVDF can be processed with many types of screw, like polyolefine type or progressive screw for example, but the following have shown good results :

Characteristics	Values
feeding	15 to 20 D
compression	3 to 0.5 D
metering	7 to 5 D
total length eg :	25 D 15 D - 3 D - 7 D 18,5 D - 0,5 D - 6 D (only short compression section for long feeding sections)
compression ratio	2,5 to 3 (ideally 3)
pitch	diameter
front angle of screw	60°

- in general, stagnation zones and excessive shear should be avoided (risks of formation of a gamma phase, which is a crystalline phase with lower thermal stability)
- for industrial production, the screw and barrel material should resist both corrosion (HF) and abrasion
- if different materials are used for the screw and the barrel, the screw hardness should be lower than the barrel hardness

- the following combinations can be suggested :

SCREW :	BARREL :
(nitruated steel)	(nitruated steel)
Nickel plated (Kanigen®)	Nickel plated (Kanigen®)
nitruated steel/Stellite 6	(nitruated steel)
nitruated steel/Stellite 6 (H)	Xaloy 306
nitruated steel/Stellite 6 (H)	Xaloy 800
hard chromium plated/stellite 6	Xaloy 800 or Bernex
Hastelloy C 276	bimetallic Bernex
Hastelloy C 276/Xaloy 330	Xaloy 309 (no Fe present!)

1.3. HEAD - DIE - CORE

- a pressure gauge (to check the backpressure) and temperature gauge (to check the melt temperature) should preferably be installed at the head
- screen packs and breaker plate can be used in order to impose some additional backpressure and provide some filtering (for high purity applications)
eg 40/80/40mesh / breaker plate
- however, take into account that excessive backpressure can lead to creep of the polymer into the extruder connections
- die/core design : in general, rounded angles are recommended

draw down ratio	1 to 1,15
draw dahn balance	0,96 to 1
die cone	13 °
core cone	11 °
length of cone	6 times core diameter
length of land	> 15 times die gap (could be shorter for large diameter pipe, high thickness or for low line speed)

- for small tubes (small internal diameter, small thickness), a slight internal air pressure can be applied through the center of the core tube in order to maintain the inner diameter and the requested thickness of the tubing or to prevent collapse of the tubing
- for the die and core material, Duranickel 301, Hastelloy, a hard chromium plating or a nickel plating are recommended for the pieces in contact with the polymer. The use of low Iron tooling is specially usefull with VF2-CTFE copolymers, which show more sticking on the tooling
- the surface finishing of the die and core material is of great importance to control the roughness of the final tube

1.4. VACUUM SIZING

- an external vacuum calibrator is recommended (vacuum which can be controlled and which is applied at the entrance and over the complete bath). Typical values 0.5 bars are obtained.
- the cooling water has to be free of impurities and contaminations
- the tube has to be completely immersed
- the possibility to increase the temperature of the water bath can be beneficial to control the crystallization
- the entrance angle has to be smooth
- too tight sealing joints can cause vibrations, which cause marks in the tube
- the vacuum pump has to be connected in a flexible way in order to avoid vibrations
- taking into account the shrinkage of PVDF, the calibrator diameter has to be 4 % in excess of the requested final tube diameter for low speed extrusion. When the speed increases, the excess has to be greater.

1.5. TAKE OFF

- avoid vibrations
- avoid compression of the tube

2. OPERATING THE EXTRUDER

2.1. SET UP

- virgin grades pellets do not have to be dried
- a clean extruder should be used in order to avoid all contaminations

2.2. TEMPERATURE PROFILE

zone 1	180 to 200 °C
zone 2	200 to 220 °C
zone 3	210 to 230 °C
zone 4	210 to 230 °C
breaker	220 to 240 °C
head	220 to 240 °C
die	220 to 240 °C

2.3. STOPPING - RESTARTING

- when stopping extrusion of SOLEF ® PVDF homopolymer (SOLEF® 1000 and 6000 series) during

0 - 0,5 hours :	maintain the set temperatures
0,5 - 2 hours :	decrease the set temperatures to 180 °C
> 2 hours :	stop the heating

- before restarting, heat to the normal set temperatures
- start again extrusion when the melt temperature is at > 200 °C for SOLEF ® PVDF 1010 (standard homopolymer extrusion grade)
- please consider that VF2-CTFE (SOLEF ® 30000 and 60000 series) copolymers have a lower thermal stability, and hence should be purged with homopolymer before stopping the extruder. Do not hesitate to contact SOLVAY CENTRAL LABORATORY for more detailed information

2.4. CLEANING

- at the end of an extrusion program (or if problems of degradation would occur), purge with a thermostable, highly viscous, pure polymer, which processing is compatible with PVDF :

eg HDPE (Eltex HDPE 5920), PMMA, PP or highly stabilized special PVC compound (Benvic® ER 499)

- never burn PVDF wastes
- do not clean pieces in a salt bath
- physical cleaning of screw and barrel has to be done (use brass tooling)
- physically cleaned screw, die and core can be put into an ultrasonic bath, filled with dimethylacetamide and heated up to 60 °C (immersion time : 1 hour) under sufficient venting (due to toxicity of solvent).

3. SAFETY

3.1. PRESSURE

Melt pressure depends greatly of viscosity and equipment (filters, die dimensions, etc.). Typically, melt pressures between 100 and 200 bars are observed during conventional extrusion.

3.2. DECOMPOSITION

- in thermogravimetric analysis (TGA), progressive degradation of PVDF homopolymer is noted from 380 °C onwards. Nevertheless, do not forget that actual processing conditions can be much harsher.
- the degradation reaction is autocatalytic
- HF will be released
- with VF2-CTFE copolymers (30000 and 60000 series) HCl will also be released. These copolymers hence show a lower thermal stability
- the decomposition temperature can be lowered by light metals, such as Ti, B, Al, ..., therefore care should be taken to avoid all contaminations coming from
 - additives
 - such as pigments (TiO₂!)
 - such as glass beads and fibers (presence of TiO₂, B₂O₃, Al₂O₃)
 - contact with light metals such as Ti, Al, B, ...
 - P₂O₅, Na₂O₂, ...
 - impurities (of previous processing for instance), greases and silicones

please contact SOLVAY in case you have any doubt.

3.3. SAFETY MARGING

- since the standard melt temperatures of PVDF do generally not exceed 250 °C during extrusion and since decomposition does not take place below 350 °C, a safety marging of roughly 100 °C is observed for homopolymer
- this safety marging is reduced with VF2-CTFE copolymers. Nevertheless, these products are processed without problems at temperatures lower than 250°C

NOTE

All the information given in these pages can only be considered as examples for processing of SOLEF. It cannot be considered as specifications or as a guarantee for successful extrusion of SOLEF® PVDF.