POLYPROPYLENE-NUCLEATION PROCESS EXPLAINED

Polypropylene general information :

Polypropylene is a semi-crystalline polymer used in wide range of applications like automotive, appliances, piping, and packaging. Isotactic Polypropylene was discovered by G. Natta in the '50s. Spheripol technology developed in 1988 allows today production of polypropylene with isotacticity index higher than 97%. Recent developments in Metallocene catalysis allow to reach almost 100% of Isotacticity index. Isotacticity increases crystallization, flexural modulus, hardness and transparency. Isotacticity decreases impact resistance and permeability. Isotactic polypropylene can crystallize under 3 forms

Alpha Phase : This a-phase is the most stable and the most known. The crystals are monoclinic.

Beta Phase : This phase is metastable and the crystals are pseudo-hexagonal b-phase is mainly found in block PP.

Gama Phase : This phase is also metastable with triclinic crystals.

Nucleating agents :

It is recognized that start-up point of crystallization of polymers are small germs (little particules) naturally included in the melt like catalyst residues, impurities, dust, etc. It is then possible to modify and control crystalline morphology by addition of " artificial " germs introduced in the polymer melt. This operation is called NUCLEATION.

Nucleating agent has to fulfill the following requirements :

- Insoluble in the polymer
- Melt point higher than the polymer
- Particle size between 1-10 microns
- Homogeneous dispersion

• The role of these nucleating agents is to improve physical and mechanical properties of finished parts.

• Nucleated PP molding formulations are often used for production of thin-walled injection molded parts (< 0.4 mm) where stiffness is required.

• In some cases, cycle-time can be shortened by 30%. Nucleating agents are also used as clarifier for films, sheets and molded parts, particularly for random PP copolymers.

Mineral nucleating agents

It has been proved that carbon black, silica, koalin or talc can be used as nucleating agents for PP. However these minerals are much less efficient than organic compounds. Moreover, it seems that these minerals are essentially efficient for homopolymers, slightly active for block PP copolymers and completely inactive for random PP copolymers.

Organic nucleating agents

Sodium, lithium or potassium salts

Efficiency of organic molecules as nucleating agent of PP has first been reported 20 years ago. One of the first nucleating agents used to improve physical properties of PP is sodium benzoate. Potassium benzoate and b-sodium naphtenoate are also efficient by increasing crystallization temperature and reducing spherulite diameter.

For optimal effect, average particle size (D50) has to be lower than 1 micron with good consistency. This is the reason why high shear is required for dispersion of sodium benzoate. A lubricating agent like glycerol monostearate (GMS) is recommended to reach the required homogeneity during processing.

Sorbitol Derivatives

Due to their very high efficiency, sorbitol derivatives are today the most commonly used agents used for PP. Some of the products are listed as per their timeline of use and are commonly classified as 1st / 2nd / 3rd generation Clarifying agents.

DBS	1,2,3,4 - dibenzylidene sorbitol
MDBS	1,2,3,4 - di-para-methylbenzylidene sorbitol
EDBS	1,2,3,4 - di-para-ethylbenzylidene sorbitol
DMDBS	1,2,3,4 - di-meta, para-methylbenzylidene sorbitol

Efficiencies of different sorbitol derivatives on random PP copolymer (3% ethylene content) are summarised in table 2.

Nucleating agent	Tcrist (°C)	Crystallinity (%)	Haze (%)	Flexural Modulus (N/mm^2)
Virgin PP	104.0	37.5	42	1050
Na Benzoate	115.0	40.0	33	1100
DBS	117.0	41.0	18	1200
MDBS	119.5	41.5	9	1300
EDBS	119.0	41.2	14	1250
DMDBS	124.0	41.7	6	1300

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