AL-PTBBA

(Nucleating agent for polypropylene)

Oxalis Chemicals Ltd.

Agent in Korea : Sischem Corp. (T: (02) 508-3812)

I. Overview on Nucleating Agents

1. Additives for PP

- Plasticizers : DOP / DODP / ODP / TOP / TCEP / DPA
- Antioxidants : BHT (IONOL) / IRGANOX 1010 / DLTP「YOSHITOMI」
- Nucleating Agents :
  - To improve rigidity : AL-PTBBA / AL-PTBBA 500 / NA-11, 18, 21 / Na-Benzoate / Talc, etc.
  - To improve transparency : Millad-3988 / NU-500 / NC-6 / Uniclear 2000, etc.
- Lubricants : Polyethylene Glycol / Wax / Higher Fatty Acid Glycerine Ester
- Solvents : Shellsol 71
- Pigments : Coloring Agents
- Others : UV Stabilizers / Antistatic Agents / Fire Retardants / Organic Blowing Agents

2. Nucleating agents for PP

PP crystallization begins at crystallization sites. Nucleating agents increase the number of crystallization sites in a polymer, resulting in an increase in the overall crystallization rate and a decrease in the spherulite size [1]. As a result, Nucleating agent improve the impact strength, tensile elasticity modulus, tensile strength and clarity of PP. Nucleating agents also increase crystallization rate that decrease cycle times [2].

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[1] Y.P.Khanna, Micromolecures, 26, 3696-3643 (1993)

[2] T.L. Smith, et.al. Journal of Applied Science, 52, 591-596 (1994)

Types	Main Effects	Major Products	Main Applications
Dispersion Type (No melting point)	• Improved rigidity (Tensile strength, Flexural modulus) • Enhance crystallization temp. (Shorter cycle time)	• AL-PTBBA • NA-11, 18, 21 • Na-Benzoate • Talc	• Car parts (Bumper, Console box) • Electrical appliance (Refrigerator, Washing machine)
Dissolution type (200-250°C melting point)	• Improve transparency	• Millad 3988, 3940 • NC-4, 6 • EC-1	• Container of clothes • Medical implement (Injection syringe)

Classification	Supplier	Advantage	Disadvantage	Price *
Metal Salts of Carboxylic Acids				
AL-PTBBA	JCT (Shell)	high cry.temp., rigidity	FDA (Oct 2004 ?)	cheap
Na-Benzoate	Many			very cheap (50%)
Phosphates				
NA-10	Asahi Denka	high cry.temp., rigidity	compatibility with PP	expensive (300%)
NA-11	Asahi Denka	transparency high cry.temp., rigidity		very expensive (4-500%)
NA-18	Asahi Denka	NA-11 (50%) + Mica ?? (50%)		expensive (130-150%)
Alkyl Benzylidene Sorbitols				
Methyl B.S.**	Milliken (Millad 3988, 3999, 3940) New Japan Chemical (Gel A11, MD (2nd Gen), MX (3rd Gen)) Mitsui Chemical (NC-4, 6)	transparency	smell	expensive (320%) (160%) (230%)
Ethyl B.S.	Mitsui Chemical	transparency	smell	expensive
B.S.	EC-Chemical	transparency	smell	expensive
Others				
Talc	Many			very very cheap

II. AL-PTBBA

1. Production

Location : Dainippon Ink and Chemicals – Kashima Factory (500 mta Capa.)

Flow : Receiving Feedstock → Quality Check → Feeding → Reaction → Washing → Drying → Crushing → Product Quality Check → Charge into Bags → Storage

2. Supplier

Oxalis Chemicals Ltd. (Est. 2019 Apr)

(former JCT : jv. between Shell Chemicals Japan Ltd. and Uyeno Group)

3. Typical Properties

Trade name	: AL-PTBBA
Chemical name	: Bis(p-tert-butylbenzoic acid) hydroxy aluminium
CAS No	: 13170-05-3
Mol formula	: $C_{22}H_{27}AlO_5$
Mol. weight	: 398
Decomposition temp.	: 250 ^oC above
Density	: 1.2 g/cm^3 ($20^oC/4^oC$)
Container	: Kraft Paper Bags with PE Liner
Net Weight	: 10 Kg/bag
Specifications	:

Test Item	AL-PTBBA Standard (#300)	AL-PTBBA#500 Fine (#500)	Test Method
Aluminium Content (%)	6.20 – 6.60	6.20 – 6.60	DIC KD-000-0 *
Water Content (%)	0.6 max	1.0 max	JIS K0067-1966 (2-1) or DIC KD-306-0
Ash Content (%)	12.4 – 16.0	12.4 – 16.0	JIS K0067-1966 (2-4) or DIC KD-542-0
Sulfate Ion (%)	1.0 max	1.0 max	JIS K8001-1985 (5-15-1) or DIC KD-427-0
Particle Size (%)	98.0 min (45μ m)	99.9 min (25μ m)	JIS K0069-1966 (3-2) or DIC KD-532-0
Appearance	White fine powder		Visual Inspection

* Test method utilizing XO (xylenol orange) as a chelating indicator.

4. AL-PTBBA : Standard vs. Fine Grade

Particle size of AL-PTBBA (FINE) is smaller than AL-PTBBA (STANDARD).

In some cases, because of good dispersibility, AL-PTBBA (FINE GRADE) is most effective agents for improving flexural modulus and flexural strength.

Fig.7&8 show the flexural modulus and flexural strength of impact PP nucleated with AL-PTBBA (FINE),AL-PTBBA (STANDARD) and NA-11.

(Resin:J3054HP(Idemitsu PC;Impact PP))

Fig.7

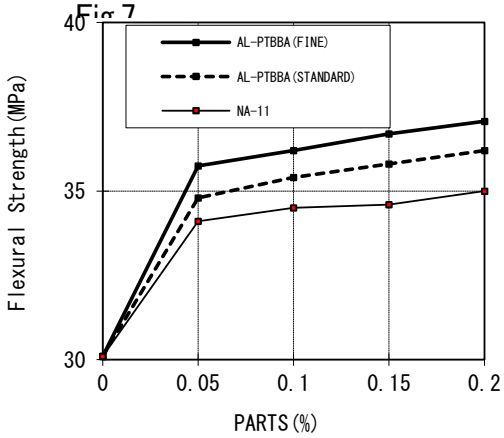
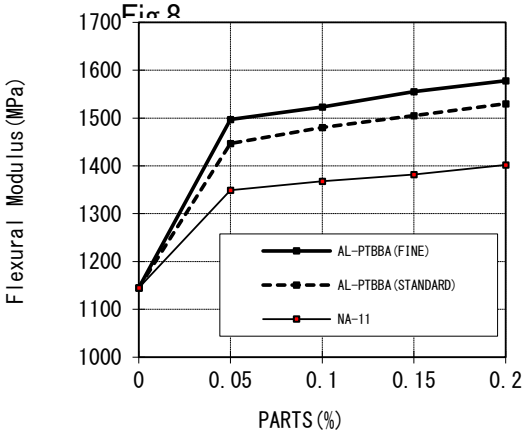
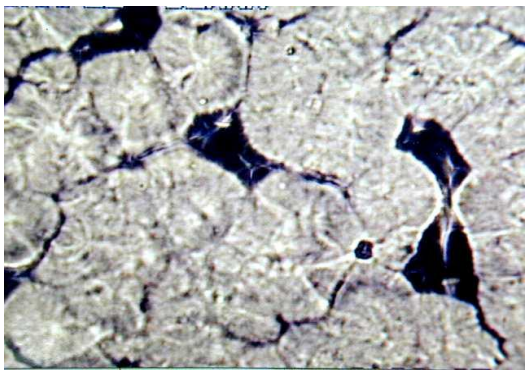


Fig.8

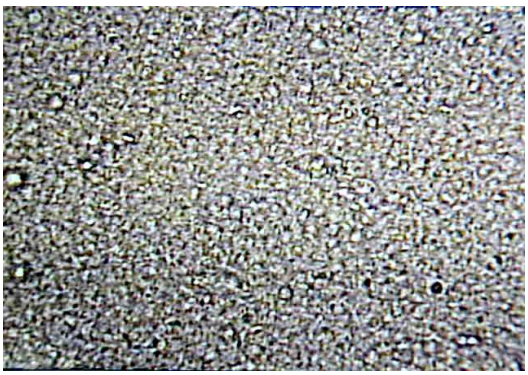


5. Advantages of AL-PTBBA

- Improve the Rigidity (injection moulding) and Transparency (injection moulded articles and sheets)
- Higher crystallization temp. enables processors to shorten cooling time
- Superior to its cost / performance competitiveness



Without Nucleating agents (100℃)



Nucleated with AL-PTBBA(120℃)

* Photo:Crystal of PP(spherulite)x2000

6. Main Application of AL-PTBBA

	none-Filler	with Filler
Homo-polymer	Food Cup (sheet) Battery Case (Car) Connector (Car)	
co-polymer		For Car Parts Bumper, Door Trimming, Console Box, Inside Wall, Instrument Panel, Connector, Lamp Housing For Electric parts and others Refrigerator inside, Speaker Box, Cap for bottle, Body of rice cooker

7. Certificates

- DIC Kashima Factory :
  - ISO 9001 / 9002 issued by Japan QAO (# JMI-0031 July 3. 1992)
  - ISO 9001 issued by BSI (# FM 21423 Sep 2. 1992)
  - ISO 14001 issued by Japan QAO (# JQA-E-80005 Jul 10. 1996)
- AL-PTBBA
  - JHOSPA (# [B] NL–0088 October 4. 1989)
    (Japan Hygienic Olefin and Styrene Plastics Association)
  - REACH Pre-registration No. : 05-2117930231-57-0000 (December 15, 2008)
  - FDA Certificate ((FCN) 001171 June 28, 2012)

III. Technical Information

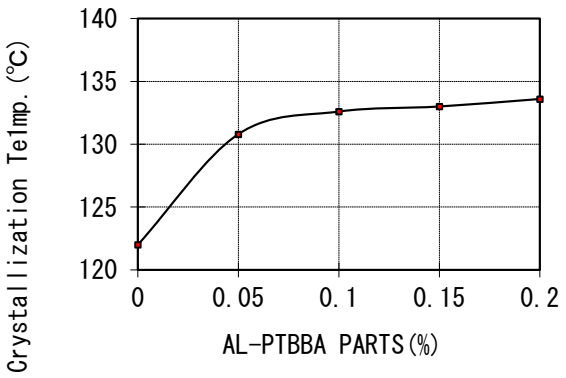
1. Crystallization temperature

The higher crystallization temperature of polypropylene, obtained by adding nucleating agents, makes it possible to reduce cooling time for many moldings. As a result it reduce molding cycle times.

Fig.1 shows the crystallization temperature of homo and random PP nucleated with AL-PTBBA. Crystallization temperature has risen approx. 10 or 15℃

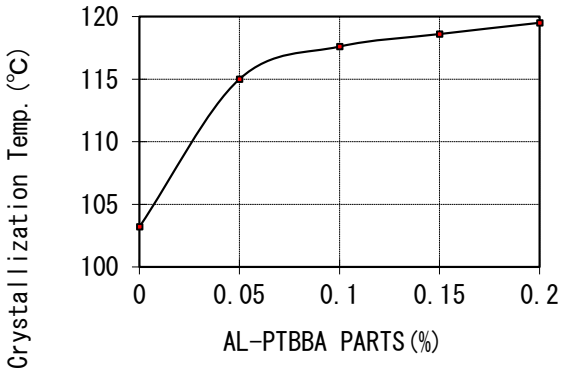
Crystallization Temperature
(Homo PP:W101 Sumitomo Chemical)

Fig. 1



Crystallization Temperature
(Random PP:W531 Sumitomo Chemical)

Fig. 2



2. Flexural modulus and HDT

The most marked feature of polypropylene articles nucleated with AL-PTBBA is an improvement of flexural modulus and HDT(Heat distortion temperature).

Table 1 shows the test result. The flexural strength and modulus of PP nucleated with AL-PTBBA are approx.10% higher than that of neat resin. And HDT is also 5 degrees higher than neat resin.

Table 1. Properties of PP(Impact copolymer) Nucleated with AL-PTBBA

AL-PTBBA parts			0%	0.1%	0.2%	0.3%
Test items	unit	Test method				
Flexural strength	MPa	ASTM D790	32.4	34.3	35.3	35.3
Flexural modulus	MPa	ASTM D790	1138	1226	1265	1285
HDT(0.45MPa.)	℃	ASTM D648	105.2	109.4	109.7	110.8

Resin:Impact copolymer (AH664A:Sumitomo Chem.)

Flexural strength and Flexural modulus of PP nucleated with AL-PTBBA are also enhanced. Table 2 and 3 show the increase of Flexural performance and crystallization temperature of homo-polymer and random-copolymer.

Table 2. Properties of PP(Homo polymer) Nucleated with AL-PTBBA

AL-PTBBA parts			0%	0.05%	0.10%	0.15%	0.20%
Test items	unit	Test method					
Flexural strength	MPa	ASTM D790	38.1	44.1	44.1	44.7	45.2
Flexural modulus	MPa	ASTM D790	1332	1641	1636	1664	1683
Crystallization temp.	°C	ASTM D648	122.0	130.8	131.9	133.0	133.6

Resin:Homo polymer (W101:Sumitomo chem.)

Table 3. Properties of PP(Random copolymer) Nucleated with AL-PTBBA

AL-PTBBA parts			0%	0.05%	0.10%	0.15%	0.20%
Test items	unit	Test method					
Flexural strength	MPa	ASTM D790	23.2	26.6	27.1	27.3	27.5
Flexural modulus	MPa	ASTM D790	727	863	890	898	904
Crystallization temp.	°C	ASTM D648	103.2	116.7	117.6	118.3	119.5

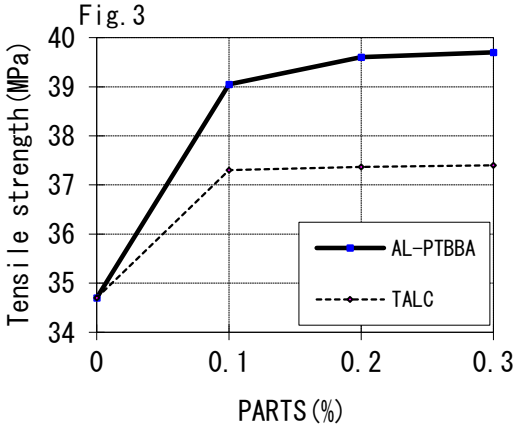
Resin:Random copolymer (W531:Sumitomo chem.)

3. Tensile modulus

Tensile strength and modulus of Homo-PP nucleated with AL-PTBBA are enhanced. And AL-PTBBA is more effective than TALC.

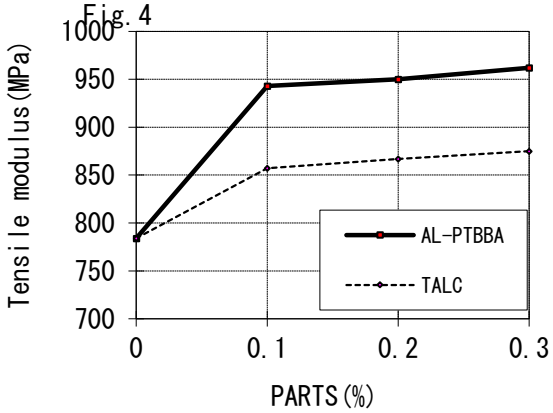
Tensile strength of Homo PP

Fig.3



Tensile modulus of Homo PP

Fig.4



4. Comparison with other nucleating agents

Flexural strength and modulus of Impact PP nucleated with AL-PTBBA, NA-11 and TALC are measured. It is apparent from the data AL-PTBBA is most effective agents for improving flexural strength and modulus.(Resin:J3054HP(Idemitsu PC;Impact PP))

Fig. 5

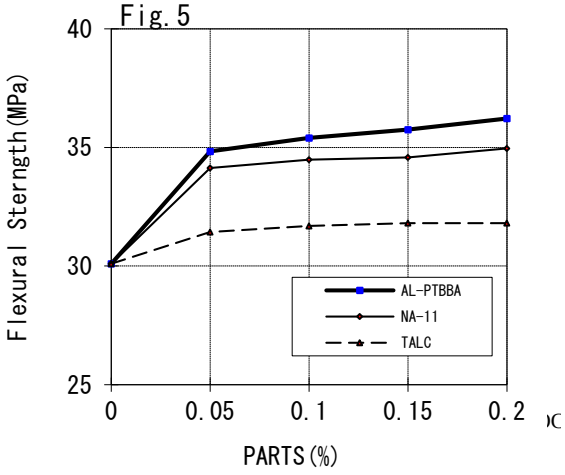


Fig. 6