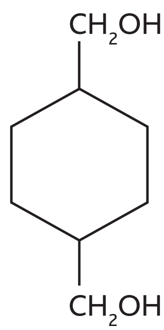
Eastman™ CHDM-D in thermoset polyester pelletized molding compounds



Eastman™ CHDM-D

Introduction

Eastman™ CHDM-D is an excellent intermediate for the synthesis of crystalline resins for pelletized molding compounds. Basic resin synthesis, compounding, molding, and physical property information are given in this publication.

CHDM products

Eastman™ CHDM-D is a reactive diol having a cycloaliphatic structure containing two primary hydroxyl sites. The product is a mixture of *cis*- and *trans*-isomers. For best processing results, CHDM-D should be heated to 65°C (149°F) until entirely molten and agitated to ensure homogeneity. Eastman™ CHDM-D90 is a solution of 90% CHDM-D and 10% water. CHDM-D90 is a liquid at normal room temperature.

Either glycol product may be used as an intermediate to make crystalline resins for pelletized molding compounds.

Synthesis of Eastman™ CHDM-D/FA resin

One resin formulation developed for use in a pelletized molding compound is an unsaturated polyester prepared from CHDM and fumaric acid (FA). A typical reactor charge and reaction conditions for the resin are given in Table 1. This polyesterification synthesis is very rapid without the use of a catalyst.

Table 1 **Composition**

Reactants	Moles	Grams
Eastman™ CHDM-D	4.56	658
Fumaric acid	4.27	496
	Total charge	1,154
	Water loss	-154
	Yield	1,000

Reaction conditions and physical constants	
Glycol excess, mole %	7
Up-heat time, hours to 200°C (392°F)	1.5
Reaction time, hours @ 200°C (392°F)	2.0
Final acid value, mg KOH/g resin	20
Molecular weight (vapor pressure osmometry)	2,000

The neat Eastman™ CHDM-D/FA polyester exhibits a high melting point range (120°–125°C [248°–257°F]). The polyester is soluble in common vinyl monomers or solvents above its melting point but does not dissolve at room temperature. A solution of CHDM-D/FA polyester in vinyl monomer at 125°–130°C (257°–266°F) becomes a solid on cooling below 120°C (248°F). The melting point of the solid solution is a function of the "crystallinity" of the polyester, which is dependent on its composition.

Table 2 shows the effects of resin formulation on the melting point of the neat polyesters and the consistency of the solid resin/monomer solutions. These polyesters were prepared from Eastman™ CHDM-D and various other intermediates and dissolved in styrene at 150°C (302°F). On cooling to room temperature, the solid resin solutions were ranked with respect to their consistency.

Table 2 **Melting point of neat polyesters and consistency of solid resin/monomer solutions (50% styrene)**

Formulation	Melting point range of neat polyesters °C (°F)	Consistency of resin/monomer solutions
Eastman™ CHDM-D/FA	>120 (>248)	Hard, brittle
Eastman™ CHDM-D/MA/FA (1:3)[a]	100–110 (212–230)	Hard
Eastman™ CHDM-D/MA/FA (1:1)[a]	80–90 (176–194)	Hard wax
Eastman™ CHDM-D/IPA/FA (1:3)[a]	95–105 (203–221)	Wax
Eastman™ CHDM-D/Eastman NPG™ glycol/DMT/FA (3:1)[b]; (1:3)[a]	40–80 (104–176)	Soft wax

[a]*Acid ratio*

[b]*Glycol ratio*

All acid or glycol modifications of the Eastman™ CHDM-D/FA formulation resulted in lower-melting neat polyesters and resin/monomer solutions that solidified more slowly.

Preparation of pelletized molding compounds

Various procedures may be followed to prepare molding compounds. A method used by Eastman's laboratory was extrusion compounding followed by dieface pelletizing. The Eastman™ CHDM-D/FA polyester polymer was ground to a fine powder using a laboratory blender. A dry blend was then prepared by tumble mixing or low-shear mixing the following: CHDM-D/FA, filler, mold release, peroxide initiator, monomer, inhibitor, low-shrink additive, and chopped-strand glass fiber. The compound formulation used for laboratory development work is given in Table 3.

Table 3 **Thermoset pelletized polyester molding compound formulation**

Formulation	Parts by weight
Eastman™ CHDM-D/FA polyester	13.9
Diallyl phthalate	7.5
Eastman™ CAB-551-0.01/pigment chips (50% CAB)[a]	2.4
Dicumyl peroxide (40% on $CaCO_3$)	0.6
Zinc stearate	1.1
Fumed silica	0.5
Calcium carbonate filler	59.0
Chopped glass strands[b]	15.0
Eastman™ hydroquinone (HQ)	125.0 ppm
p-benzoquinone (p-BQ)	125.0 ppm

[a]*Eastman™ cellulose acetate butyrate*

[b]*Owens Corning*

The dry blend was fed into a heated extruder having a zoned temperature control system. The extruder melted the polyester resin, blended it thoroughly with the other ingredients, and forced the molten mixture into a strand die.

Temperatures necessary to melt the crystalline polyester (125°–130°C [257°–266°F]) should be used; however, care should be taken to avoid higher temperatures that could rapidly decompose the peroxide initiator and cure the resin in the barrel. The strand die should be slightly below the melting point of the crystalline resin so that hardening of the extrudate begins before chopping the strand in the pelletizing system. For the Eastman™ CHDM-D/FA resin, a die temperature of about 120°C (248°F) was used. The modified resins would require somewhat lower temperatures depending on the relative hardness of the resin/monomer solution (see Table 2).

Injection molding pelletized compounds

Processing parameters for injection molding pelletized compounds should be carefully controlled. Suggested trial values for reciprocating-screw injection machines follow:

Table 4

Barrel temperature	120°–130°C (257°–266°F)
Mold temperature	150°–160°C (302°–320°F)
Screw speed	Slow–medium
Back pressure	Low
Injection speed	Fast
Injection pressure	High
Hold time	60–90 s (to cure)

Injection molding a dry-blended compound

An alternative technique for injection molding is to prepare dry-blended compound from the ground polyester, fillers, monomers, release agents, etc., using the formulation in Table 3. The dry blend can be fed directly into a reciprocating screw in an injection molding machine without going through a separate pelletizing process. Suggested machine parameters are the same as for the pelletized compound.

Molded test specimens based on a dry-blended molding compound using Eastman™ CHDM-D/FA were obtained and physical properties were determined. The results follow:

Table 5 **Physical properties of a cured, dry-blended molding compound based on Eastman™ CHDM-D/FA resin**

Tensile strength, MPa (psi)	34.5 (5,000)
Tensile modulus, MPa (psi x 10^5)	1,468 (2.1)
Flexural strength, MPa (psi)	86.9 (12,600)
Flexural modulus, MPa (psi x 10^5)	1,331 (1.9)
Notched Izod impact strength, ft-lb/in. of notch	2.8

Application

Resins based on Eastman™ CHDM-D are suggested for dry-pelletized molding compounds where good heat stability, electrical properties, and corrosion resistance are needed.

Summary

Eastman™ CHDM-D used in the development of dry-pelletized molding compounds can provide:

- Rapid resin synthesis
- “Crystalline” resin characteristics
- Formulation flexibility
- Nonblocking characteristics
- Excellent thermal stability
- Low water absorption
- Very good electrical properties and corrosion resistance

For more information on Eastman™ CHDM, visit www.eastman.com.

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