

Copolymerized Silane PEX Technology A New Innovation for Producing PEX Pipes

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Abstract

For nearly three decades two technology processes have been available for production of PEX piping by moisture induced silane crosslinking (PEX-b). The first technology, the Sioplas™ process, was invented and patented by Dow Corning. This process requires extruding pipe from a reactive compound composed of silane groups which have been grafted onto polyethylene (PE) polymer chains by the addition of organic peroxide in an off-line compounding process. The second technology, the Monosil™ process, was developed by Maillefer as a one-step-process where all of the components of PE base resin, additives, peroxide and silane are grafted in a specialized compounding extruder which also extrudes the pipe in-line. This paper describes a new innovative technology process that offers the PEX pipe industry a third choice in materials for producing PEX-b piping. The **Vinyl Silane Copolymer** or Visico™ process, invented and patented by Borealis AS, copolymerizes the silane directly into the PE polymer chain during the polymerization process. This ensures an excellent homogeneous distribution of the reactive silane sites and avoids the heavy use of organic peroxides needed by the other PEX-b technologies to graft the silane component. The resulting product offers greater process stability and higher purity for superior taste & odor performance in PEX piping. All of these technologies require post treating the pipe with steam or hot water to induce the crosslinking reaction and produce PEX-b pipe although the new Borealis Ambicat™ catalyst system for MDPE PEX-b radiant heating pipe enables crosslinking to take place during pipe storage at ambient temperatures by absorbing moisture from the surrounding air in the warehouse or yard.

INTRODUCTION

PEX, or crosslinked polyethylene, tubing is one of the most durable, cost-effective and easy-to-install plumbing pipes around the world. For decades it has been used successfully for residential and commercial hot & cold potable water supply lines, radiant floor heating, snow melting applications and radiant cooling for ice rinks or refrigerated storage. PEX tubing is



also compatible for use in conjunction with legacy piping systems of steel or copper simply by selecting the correct fittings and adapters. PEX piping offers good flexibility that allows the pipe to expand and contract slightly to minimize the chances of pipe burst and eliminates

water hammer. This flexibility also allows it to be bent around corners without the use of fittings, allowing for faster, less expensive installation during new construction. PEX piping does not corrode as metal pipes do and is highly resistant to mineral buildup and scaling. In



addition, PEX pipe's low thermal conductivity minimizes heat loss and dramatically reduces condensation which can be a common problem of metal pipe PPI – HTD (2).

PE, already a plastic of choice for larger diameter supply mains for potable water and natural gas, gains improved high temperature, abrasion and stress crack resistance from the crosslink process used to create PEX tubing. These properties have made PEX tubing the pipe of choice for plumbing around the world, replacing copper pipe in most new housing construction. The market for PEX pipe in North America in particular has grown on the average of 25% per year over the past several years with its fastest growth in the hot & cold water segment, taking market share from copper (3).

The exceptional performance of PEX tubing comes from the durability and inert nature of polyethylene (PE) combined with the process of crosslinking, which creates intermolecular covalent bonds between the PE polymer chains Carley (4). PEX piping is typically produced using one of three methods, often referred to as PEX-a, PEX-b and PEX-c, all of which are capable of producing suitable PEX piping. The PEX-a method extrudes organic peroxide along with the PE at a temperature that is high enough to cause the peroxide to decompose and promote bonding between the polymer chains. PEX-b methods incorporate an organic silane compound along with a peroxide and metal catalyst that allows steam or hot water to crosslink the extruded tubing in a secondary process. PEX-c method extrudes thermoplastic PE piping that is exposed either inline or in a secondary process to an electron-beam radiation source which promotes crosslinking. This paper will focus on recent innovations that improve material properties and add value to the PEX-b method.

SILANE CROSSLINKING TECHNOLOGY

The PEX-b method of producing PEX piping, often referred to as silane crosslinking technology or the moisture crosslinking process, originated in the Wire & Cable industry as a means of insulating metal conductors. Moisture-induced silane crosslinking technology was designed to replace PE compounds that were soaked in peroxide and then crosslinked by extrusion through a steam or molten salt vulcanization tube. This vulcanization process is not entirely unlike some current PEX-a process technologies used to produce PEX tubing.

PEX-b has been the fastest growing segment in the production of PEX piping due to its relatively low capital investment in comparison to PEX-a and PEX-c methods. A standard thermoplastic pipe extrusion line combined with a post-extrusion hot water treatment apparatus to flush and crosslink the tubing allowed a pipe manufacturer to enter the fast growing PEX market using Sioplas™ process technology. Invented and patented by Dow

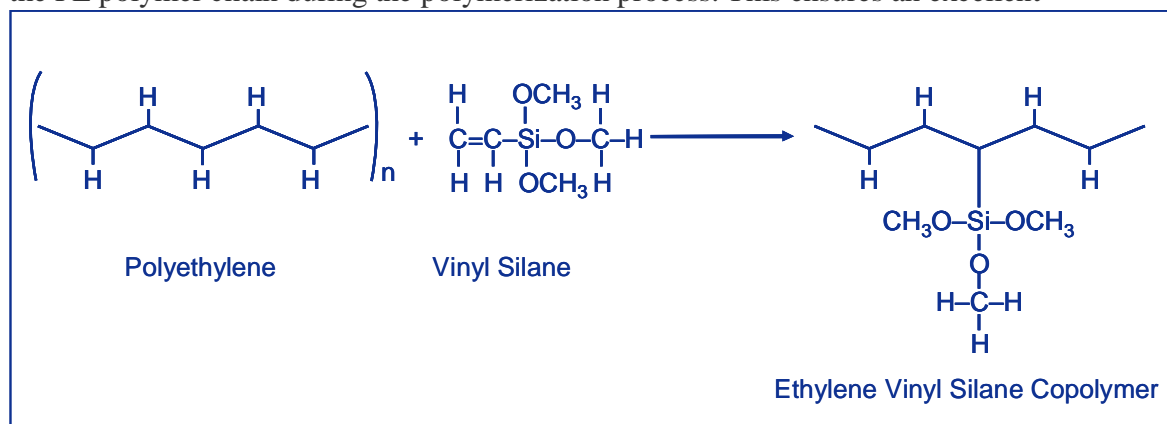
Corning in 1968, the Sioplas process extrudes tubing pipe from a reactive compound composed of silane groups which have been grafted onto PE polymer chains Ribarits (5). This is accomplished by a custom compounder who grafts the silane onto an HDPE by adding organic peroxide in a high temperature compounding, extrusion and pelletizing process. This reactive compound, when combined with a tin catalyst masterbatch and extruded into tubing by the pipe manufacturer, will the crosslink in PEX tubing after being flushed with hot water.

An alternate technology for PEX-b tubing production is referred to as the Monosil™ process and was developed jointly by Maillefer and BICC in 1974 as a one-step-process, where all of the components of PE base resin, additives, peroxide and silane are grafted in a specialized compounding extruder which also extrudes the pipe in-line Ribarits (5). Like the Sioplas technology, the Monosil technology also requires a post-extrusion treatment that flushes hot water through the pipe to initiate the crosslinking reaction.

The key to this moisture-induced crosslinking is the silane molecule that has been grafted onto the PE polymer chain. Unlike PEX-a and PEX-c technologies that forge carbon to carbon bonds during their crosslinking process, PEX-b crosslinking links PE polymer chains together by forming a “silane bridge.” Grafting places a trimethoxysilane side group [-Si-(OCH₃)₃] onto the PE chain that during crosslinking first undergoes a hydrolysis reaction during which the three trimethyl groups are cleaved and converted into methanol (3 CH₃OH). Two PE chains containing the silane hydroxyl [-Si-(OH)₃] pendants next form the crosslink site by undergoing a condensation reaction Plueddemann (6)

NOVEL MATERIALS FOR SILANE CROSSLINKING

The **Vinyl Silane Copolymer** or Visico™ process, invented and patented by Borealis AS, offers the PEX pipe industry an innovative technology improvement that is a new third choice for producing PEX-b piping. Visico technology copolymerizes the silane directly into the PE polymer chain during the polymerization process. This ensures an excellent



homogeneous distribution of the reactive silane sites and avoids the heavy use of organic peroxides needed to graft the silane component in the other PEX-b technologies. Since copolymerization brings little to no residue products to the final base compound, the resulting product offers greater process stability and higher purity for obvious benefits in pipe

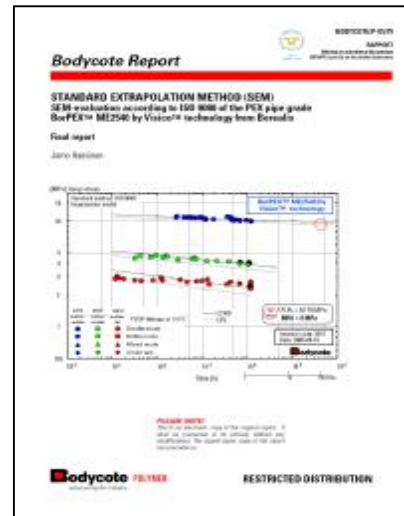
(68), 95 (203) and 110°C (230°F). In the Bodycote report on BorPEX HE2545 to the right, hydrostatic test results exhibit flat curves with very little data scatter, indicating a consistent product of uniform composition Bodycote (7).

The combination of BorPEX ME2540 combined with either Cat-MB420 or Ambicat™ MB64 when crosslinked into PEX pipe has been designed to meet the requirements of international pipe standards such as DIN 16894. This same combination is particularly well suited for use in aluminum multilayer pipes or for PEX pipes with high flexibility.

Physical Properties of PEX Pipe Made With BorPEX ME2540 and Ambicat™ MB64			
	Typical Value**	Units	Test Method
Density*	937 (0.937)	kg/m ³ (g/cm ³)	ISO 1183 (ASTM D1505)
Melt Flow Rate* (190°C/2.16 kg)	2	g/10 min	ISO 1133 (ASTM D1238)
Melt Flow Rate* (190°C/5 kg)	8	g/10 min	ISO 1133 (ASTM D1238)
OIT (210°C)	>60	minutes	EN728 (ASTM D3895)
Modulus of Elasticity	750 (10,900)	MPa (psi)	ISO 527 (ASTM D683)
Tensile Stress at Yield	17 (2470)	MPa (psi)	ISO 527 (ASTM D683)
Tensile Elongation at Break	>300	%	ISO 527 (ASTM D683)

- * Pure BorPEX HE2545 in thermoplastic state
- ** Data should not be used for specification work.

Using the Standard Extrapolation Method (SEM) according to ISO 9080:2003 we have obtained an MRS for BorPEX ME2540 of 8 MPa with an extrapolated strength of 5 MPa after 50 years lifetime at 70°C (158°F). The work covers hydrostatic pressure testing at 20 (68), 95 (203) and 110°C (230°F). In the Bodycote report on BorPEX ME2540 to the right, hydrostatic test results demonstrate very little data scatter, flat curves and consistent performance for ME2540 Bodycote (8).



EXTRUSION PERFORMANCE

BorPEX by Visico technology is designed to be processed on standard single screw extruders typically configured for grafted PEX-b. With higher line speed, cooling capacity (i.e. length of cooling baths) typically becomes the limiting factor. Using Visico™ technology offers the benefit of lower processing temperatures in the range of 170-200°C (338-392°F). By extruding pipe at cooler temperatures a greater length of pipe can be cooled in a given period of time on a given cooling section. The pipe producer will realize additional energy saving benefits in that there are lower energy costs in heating the BorPEX compound up and cooling the resulting PEX pipe down.

Many PEX pipe producers who use grafted PEX products struggle to a greater or lesser degree with plate-out, which originates from grafting residue products and overheating of compound during the pipe extrusion process. Plate-out can also result from poor additive dispersion since all of the stabilization for grafted materials is added along with the catalyst

masterbatch at the pipe extruder. Sometimes processing aids are added to coat the extrusion and die flow channels to minimize plate-out and the need to stop the line for cleaning. Thanks to a cleaner product and lower processing temperatures, extruding PEX pipe from BorPEX by Visico technology means less plate-out and longer time periods between cleaning stops. Proper dispersion of the long-term stabilizers in BorPEX products is ensured, since it has been compounded into the base resin rather than the catalyst masterbatch.

Maximum output rate of pipe production using grafted materials is typically limited by the surface quality of the extruded pipe. Grafted PEX products are somewhat prone to melt fracture, and so processing aids are often recommended by the grafted material manufacturers to overcome this phenomenon. Once again, thanks to the molecular structure and higher cleanliness of BorPEX by Visico technology, PEX pipe can be extruded at very high line speeds without melt fracture, without pre-crosslinking and without the need to use a processing aid. The first example was a test performed on extruder manufacturer Bandera's own lab line in Busto Arsizio, Italy. The single screw extrusion line was a Bandera TR65 AFTH with a 32 L/D and a maximum screw speed of 220 rpm. The Bandera "TR65" is a commonly used extruder in the PEX-b pipe industry. The extrusion screw design on this line was a common PEX-b configuration compression screw with 2 mixer zones. BorPEX ME3540 and HE2545 were both tested in 25 mm pipe using CAT MB420 to promote crosslinking. Both extrusion runs ran problem free, with maximum extruder screw speed as the limiting factor on line speed. Even at maximum speed the melt temperature was quite low for PEX-b extrusion at 185°C (365°F); however pipe surfaces were still smooth. It is not atypical to see a slight haze or dullness on smooth pipe surfaces when running at high extrusion speeds at a low melt temperature. This can easily be cured by using a heated pin in the tooling for the inner surface and a heated metal or flame ring for the outer surface. Of course, for best outer surface quality, a calibrator of underwater design should preferably be used.

		ME2540 + CATMB420		HE2545 + CATMB420	
Sample Number		1	2	3	4
Extruder					
Feeding zone	°C (°F)	30 (86)	30 (86)	30 (86)	90 (194)
Zone 1	°C (°F)	160 (320)	160 (320)	170 (338)	180 (356)
Zone 2	°C (°F)	170 (338)	170 (338)	180 (356)	180 (356)
Zone 3	°C (°F)	175 (347)	175 (347)	185 (365)	185 (365)
Zone 4	°C (°F)	180 (356)	180 (356)	190 (374)	190 (374)
Melt Temp.	°C (°F)	184 (363)	185 (365)	186 (367)	192 (378)
Pressure	Bar (psi)	149 (2160)	152 (2200)	147 (2130)	144 (2090)
Screw speed	RPM	200	214	198	211
Screw torque	%	43	41	37	36
Tooling					
Zone D	°C (°F)	185 (365)	185 (365)	185 (365)	185 (365)
Zone C	°C (°F)	185 (365)	185 (365)	185 (365)	185 (365)
Zone B	°C (°F)	190 (374)	190 (374)	190 (374)	190 (374)
Zone A	°C (°F)	200 (392)	200 (392)	200 (392)	200 (392)
Line Rate					
Output	kg/h (lbs/h)	300 (660)	320 (710)	300 (660)	300 (660)
Output	m/min (ft/min)	26.3 (86.3)	28 (91.9)	26.3 (86.3)	26.3 (86.3)

The second example involves an extrusion test run at extruder manufacturer Amut, Italy. The “5 extruder -1 tooling” extrusion line was designed to produce a small diameter five layer heating pipe with the dimension of 2 x 20mm (0.079 x 0.79 inch). The construction of the pipe was a PEX inner and outer layer bonded by 2 adhesive layers to a center core layer of EVOH for its barrier properties. The extruders had 60mm (2.4 inch) diameter and 33 L/D barrier screws.

This 5 layer pipe design is prone to building up high pressure in the tooling, due to the thin flow channels for the PEX layers and high extrusion speed. High die pressure may be a direct limit to extrusion line speed and also create backflow of the polymer melt in the extruder. This backflow will, in turn, cause frictional heat buildup that will manifest itself visibly as pre-crosslinking and plate-out in extruded pipe. The extrusion line for this experiment can typically extrude grafted PEX-b products at line speeds up to 25m/min (82 ft/min), at which point the pressure in the tooling reaches the maximum level of 300 bar (4350 psi), which limits the line’s output. Extruding BorPEX Visico ME2540 on this same line in the same pipe construction allowed an output rate of 40m/min (130 ft/min) to be reached at a low melt temperature of 178°C (352°F). At this extrusion line speed the output pressure only reached 250 bar (3630 psi). The maximum extruder speed became the limiting factor to line speed rather than the output pressure. The resulting PEX pipe surfaces were glossy even at 40 m/min (130 ft/min) without the need to add a processing aid. This example illustrates how suitable the BorPEX by Visico technology concept is for the production of PEX/aluminum multilayer pipes.

CROSSLINKING PERFORMANCE

The combination BorPEX HE2545 and Cat-MB420 is crosslinked in the traditional manner by exposing the pipe to hot water or steam (sauna). Crosslinking time will of course depend on temperature, humidity and wall thickness. A typical crosslinking time for a pipe with a 2 mm (79 mil) wall thickness when exposed to hot 95°C (203°F) water is 6 hours. Pipes intended for transporting hot & cold potable water shall preferably be crosslinked by pumping hot water through the pipe in order to wash out the silane crosslinking reaction products that are formed during crosslinking Borealis (9).

Ambicat™ catalyst technology combined with BorPEX™ ME2540 allows standard PEX monolayer tubing to crosslink under ambient conditions of temperature and humidity. Once again crosslinking time will depend on temperature, humidity levels and the wall thickness of the pipe. BorPEX ME2450 and Ambicat MB64 may also be crosslinked by exposure to hot water or steam (sauna) for accelerated crosslinking times as opposed to using a traditional tin catalyst system. Crosslinking times for a pipe with a 2 mm (79 mil) wall thickness when exposed to 95°C (203°F) hot water are typically 4 hours. Crosslinking times for that same pipe construction at room temperature conditions of 23°C (73°F) and 50% humidity are typically 10 days Borealis (10)

ORGANOLEPTIC PERFORMANCE

PEX pipe produced using Visico™ technology offers superior organoleptic performance versus pipe produced from the Sioplas and Monosil technologies. This is due to the extremely low levels of residue products formed during extrusion and crosslinking of the PEX pipe. BorPEX HE2545 has significantly better taste and odor performance while BorPEX ME2540 has the best organoleptic properties. Taste and odor for both of these products is optimized by flushing out the reaction products with hot water during the crosslinking process, however the duration of this flush is significantly less than for comparable PEX pipes produced with Sioplas or Monosil technologies.

MATERIALS HANDLING AND STORAGE

BorPEX HE2545 can be stored in a dry and clean environment at 10 - 30°C (50 - 86°F), protected from UV-light, in unopened original packaging for up to one year after production, without any significant deterioration of the quality of the material. This is a significant improvement over the Sioplas technology that mandates a base compound shelf life of 4-6 months. However, it is still recommended to follow “first in–first out” principle. BorPEX ME2540 can be stored for an even longer period of time under the above conditions, up to 18 months after production, without any significant deterioration of the quality of the material. BorPEX ME2540 does not require vacuum packaging, which makes bulk handling and bulk deliveries possible. Cat-MB420 and Ambicat™ MB64 can be stored in a dry and clean environment at 10 - 30°C (50 - 86°F), protected from UV-light, in unopened original packaging for 18 months after production, without any significant deterioration of the quality of the material. However, it is recommended to follow “first in–first out” principle.

SUMMARY

A new innovative technology process offers the PEX pipe industry a third choice in materials for producing PEX-b piping. The **Vinyl Silane Copolymer** or Visico™ process technology, invented and patented by Borealis AS, copolymerizes the silane directly into the PE polymer chain during the polymerization process. This ensures an excellent homogeneous distribution of the reactive silane sites and avoids the heavy use of organic peroxides needed by the other PEX-b technologies to graft the silane component. The resulting products offer greater process stability and higher purity for superior taste & odor performance in PEX piping.

BorPEX by Visico Technology is suitable for use in standard monolayer PEX piping as well as multi-layer pipe and injection molded parts where its lower melt viscosity offers easier processing. Visico™ technology offers the benefit of lower processing temperatures than traditional silane PEX technologies while maintaining a smooth glossy pipe surface. Pipe producers can use this to increase the output rates on extrusion lines that are cooling limited and realize additional energy saving benefits at the same time. In addition the BorPEX by Visico Technology base resins, HE2545 and ME2540, are shelf stable for storage under typical warehouse conditions two to four times longer than competitive compounds produced by Sioplas technology.

BorPEX by Visico Technology is crosslinked in the traditional manner by exposing the pipe to hot water or steam (sauna). Ambicat™ catalyst technology combined with BorPEX™ ME2540 allows standard PEX monolayer tubing to crosslink under ambient conditions of temperature and humidity. Unlike conventional silane technologies, Visico™ technology allows the incorporation of the long-term stabilization package for the resulting PEX pipe to be well dispersed in the base resin during a compounding step in its manufacture. This gives the PEX pipe producer more control over crosslinking speed by controlling the rate of addition of the catalyst masterbatch.

PEX pipe produced using Visico™ technology offers superior organoleptic performance versus pipe produced from the Sioplas and Monosil technologies. This is due to the extremely low levels of residue products formed during extrusion and crosslinking of the PEX pipe. Taste and odor for BorPEX by Visico Technology piping are also optimized by flushing out the reaction products with hot water during the crosslinking process; the duration of this flush is, however, significantly less than for comparable PEX pipes produced with Sioplas or Monosil technologies.

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