NEW TESTING CAPABILITIES FOR CROSSLINKED PE

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ABSTRACT

Since the discovery of Sioplas® technology in the late sixties by Dow Corning, PEX-b pipes processing has been progressively well established and derived into various alternatives around the same theme, e.g., Monosil®, Visico®, or Spherisil®. All these routes are calling for the reaction of a vinyl-functional monomer with polyethylene in presence of free radical initiator. Independently from the processes used for modifying the high-density-polyethylene (HDPE), the goal in PEX-b pipe fabrication is to obtain a silyl-alkoxy modified HDPE that can then be extruded into pipes of various dimensions. For both compounds producers and pipes fabricators, it is important to assess the rate of crosslinking with a high degree of accuracy and as rapidly as possible in order to verify the conformity of the material against pipes industry standard, i.e., meeting the 65-75% gel content requirement according to ISO10147 method. Using Alpha Technologies VTM (Viscosity Transition Modulus) rheometer, a method for determining rapidly the gel content and for monitoring the kinetics of crosslinking in PEX-b specimens was developed. Information's on crosslinking density were obtained within 10 minutes versus 12-24 hours using the standard ISO10147 method.

On one hand, plots of elastic shear modulus (G') against percentage strain applied to melted PEX-b compounds were recorded on a series of specimens prepared with different vinylsilane concentrations, before and after a standard curing step. Good correlation between G' values and the gel contents measured upon xylene extraction were observed.

On the other hand, monitoring the increase of G' as a function of time the PEX-b specimens were curing underwater at different temperatures was shown an extremely effective mean to determine the rate of crosslinking of the material and the energy of activation of the crosslinking reaction.

Further optimization of the method was also carried out in order to monitor the crosslinking process of PEX-b compounds within the chamber of the VTM rheometer. The ultimate target was to get access to crosslinking kinetics and to determine the gel content from PEX-b compound within 1 hour starting from the vinyl-silane-grafted HDPE pellets.

The methods developed in this study are much faster, safer, reliable and significantly more economically viable than the standard gel content measurements based upon extraction in refluxing xylene. The conclusions of the correlation between the two methods, i.e., G' and gel content, are presented and discussed within the light of the plastic pipe industry needs to continuously improve material properties (R&D perspective) and/or their product quality (Quality Control perspective).