Electron Beam Crosslinking of UHMWPE-Vitamin E Blends

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Statement of Purpose: The effects of vitamin E on crosslinking of UHMWPE by electron beam irradiation were investigated using a multifactor fractional factorial design of experiments approach. Vitamin E is a well known oxidation stabilizer for commercial polyolefin materials, and is attractive for use in implant materials due to its biocompatibility. Inhibition of crosslinking by vitamin E is a concern, since vitamin E is an efficient free radical scavenger. This investigation sought to understand the level of crosslinking that could occur under different irradiation conditions and with different levels of vitamin E contained in the consolidated UHMWPE material.

Methods: For this study, GUR 1050 and GUR 1020 UHMWPE resins were blended with 0.2 wt%, 0.5 wt% and 1 wt% Vitamin E (d/l-α-tocopherol) prior to consolidation. Each of the material combinations were compression molded into a slab and cut into bars. The bars were preconditioned and e-beam irradiated according to a five factor DOE. Factors investigated included electron beam dose levels (90 – 150 kGy), dose rates (75-240 kGy-m/min.), bar temperature during irradiation (Pre-heat, 40 – 120 °C), UHMWPE resin (GUR 1020 or GUR 1050), and vitamin E level (0.2 -1.0 weight %). Crosslinking levels were investigated by measurement of FTIR *trans*-vinylene index per ASTM F-2381, and by swelling ratio of 12.5 mm cubes after soaking in boiling p-xylene for 72 hours per method described by Shen[1].

Results/Discussion: The TVI data was fitted to a quadratic polynomial for the DOE model. The most significant factor for the TVI response was the dose level, with $p<0.0001$. The interaction of dose with vitamin E level was also significant, with $p=0.007$, see figure 1. Other significant factor effects included the material pre-heat temperature and dose rate, which had a positive correlation with TVI. The swell ratio, q, exhibited linear behavior with the factors studied. The most significant factor for the swell ratio response was the temperature of the material during irradiation ($p<0.0001$). Material temperature also had a significant interaction with the vitamin E level ($p=0.03$), see figure 2. Dose also had a significant effect and correlated negatively with swell ratio ($p<0.001$).

The trans-vinyl index is affected by the vitamin E level, but the effect diminishes with increasing dose level. A similar effect is seen for crosslink density as indicated by the swell ratio, where lower swell ratios correspond with higher crosslink density. High vitamin E levels are seen to inhibit crosslinking, but the effect of vitamin E level on swell ratio (i.e. crosslinking) diminishes with increasing material temperature during irradiation. Crosslink density levels may not correlate well with the trans-vinyl index response, since the primary affect for crosslinking level was material temperature during irradiation, while the primary affect on TVI response is dose level.

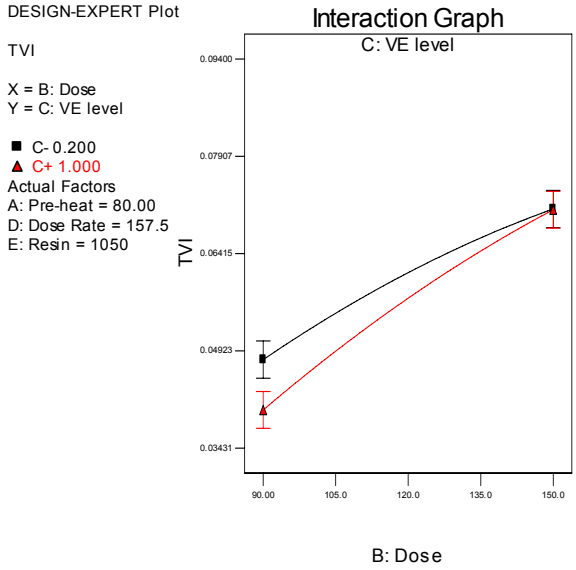

Figure 1. Dose-Vitamin E Interaction for TVI

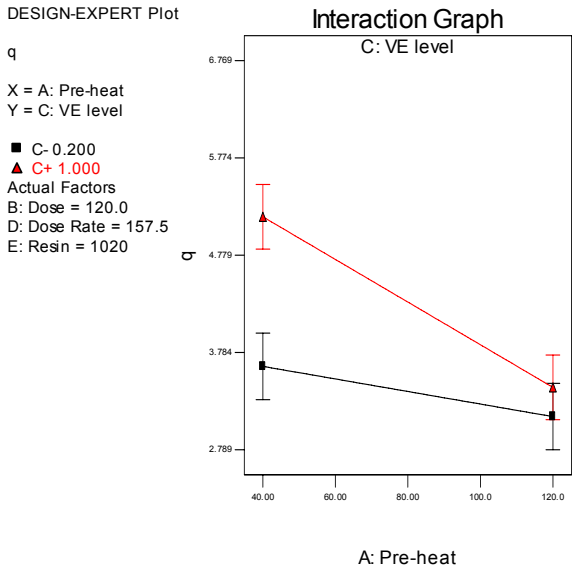

Figure 2. Temperature-Vitamin E Interaction for swell ratio (q)

Conclusions: Vitamin E inhibits cross-linking at higher loading levels, but cross-linking is possible in UHMWPE-vitamin E blends under the right conditions. Higher temperatures during irradiation promote significantly higher levels of crosslinking, especially at high levels of vitamin E. The trans-vinyl index serves as a reliable indicator of dose level, but may not be a reliable indicator of crosslink density when irradiation occurs in the presence of vitamin E.

References: 1. (Shen FW. J.Polm.Sci. 1996;34:1963-1067.)