

Studies in Irradiated UHMWPE Oxidation

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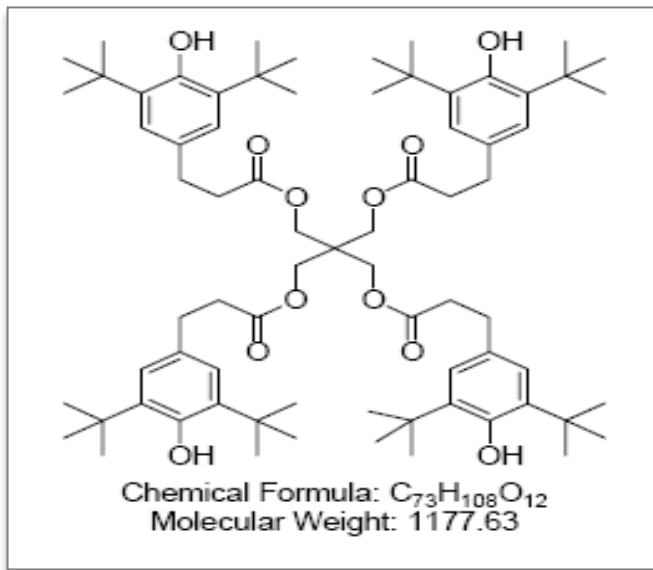
UHMWPE 2019

Objectives

- Quantify antioxidant loading in UHMWPE using FTIR Spectroscopy and Differential Scanning Calorimetry.
- Evaluate the property response for two different antioxidants as a function of concentration.
- Compare head-to-head the resulting properties of the antioxidant-compounded formulations at loadings ranging from 0.05 to 0.3% (w/w)

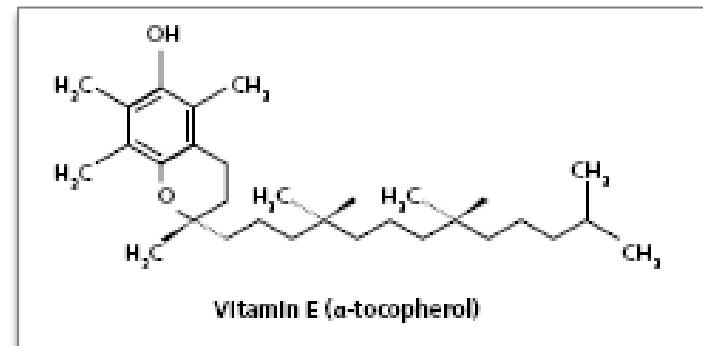
Antioxidants in the Study

COVERNOX™ (PBHP) Hindered Polyphenol



- Solid at room and body temperature.
- Melting point = 115° C.
- Extremely low extractable and leachable potential.

Vitamin E /(α -Tocopherol, α T) (Hindered Monophenol)



- Liquid at room and body temperature.
- Melting point = 3°C.
- Higher extractable and leachable potential, especially from infused formulations.

Study Matrix

UHMWPE: GUR 1020 (Celanese)

Radiation: Gamma @ 100 kGy (nominal)

Post Radiation Thermal Treatment: NONE

Antioxidants	% Loading in UHMWPE (ppm)	Test Methods
PBHP α Tocopherol	0.05 (500)	α T Index by FTIR
	0.075 (750)	PBHP Index by FTIR
	0.10 (1000)	OIT by DSC
	0.15 (1500)	GSR ASTM D2765
	0.30 (3000)	DNI Toughness

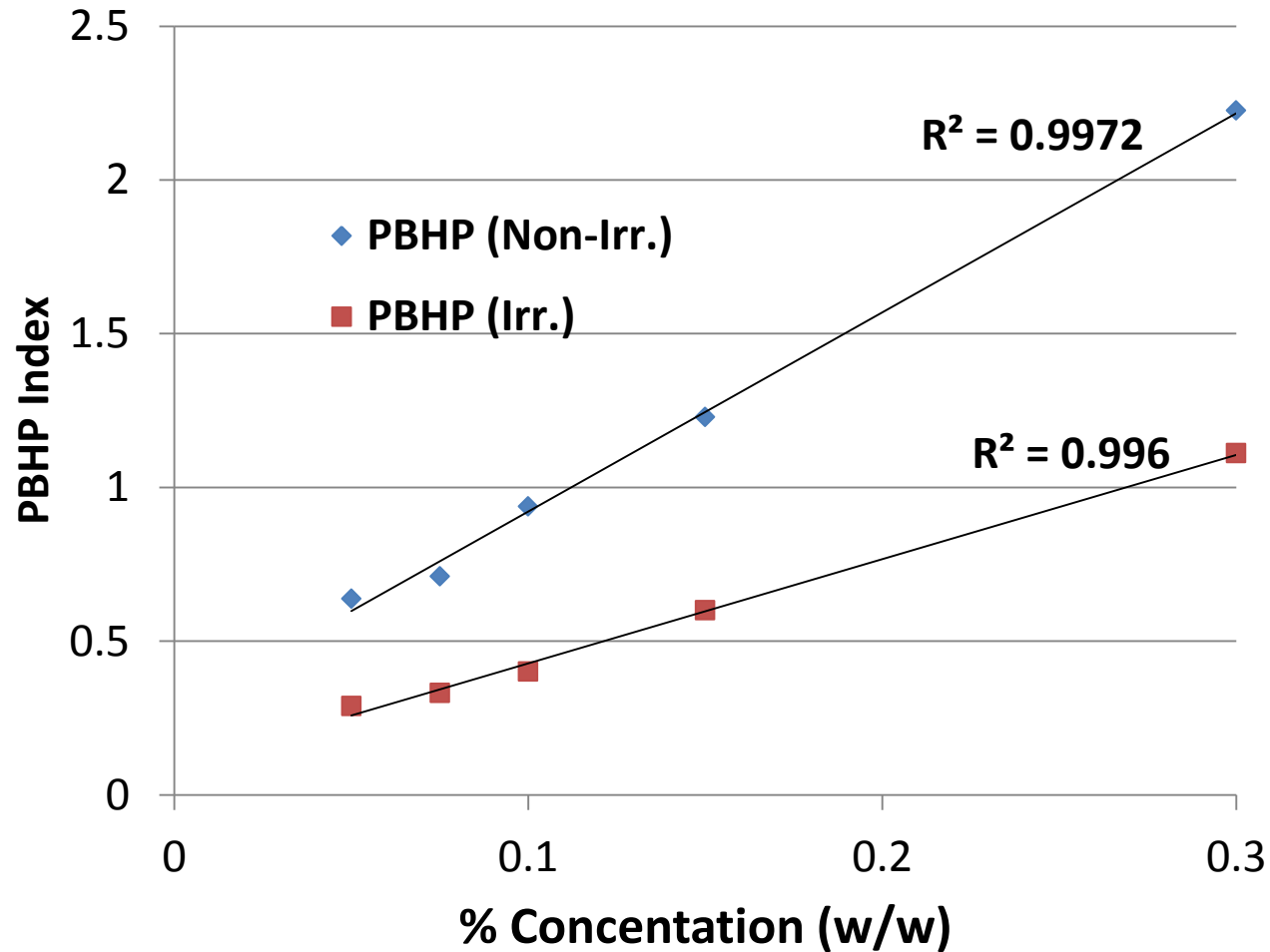
Sample Generation

- **Solvent**: Antioxidants are dissolved in HPLC grade acetone.
- **Formulations**: Formulations of both antioxidants are generated by the dispersion of the antioxidants in acetone into UHMWPE in a high shear blender at 60°C and solvent evaporation effected under vacuum.
- **Concentrations**: Appropriate, pre-calculated quantities of the antioxidant solutions are blended with 500 g. GUR 1020 UHMWPE to generate formulations at concentrations of 0.05, 0.075, 0.10, 0.15 and 0.3% (w/w) in UHMWPE.
- **Consolidation**: Compression molded plaques at 3" x 6" x 1.0" are generated in a heated hydraulic press
- **Double Notched Izod Toughness**: Test samples are generated and packaged in vacuum foil pouches and gamma-irradiated to a nominal of 100 kGy.

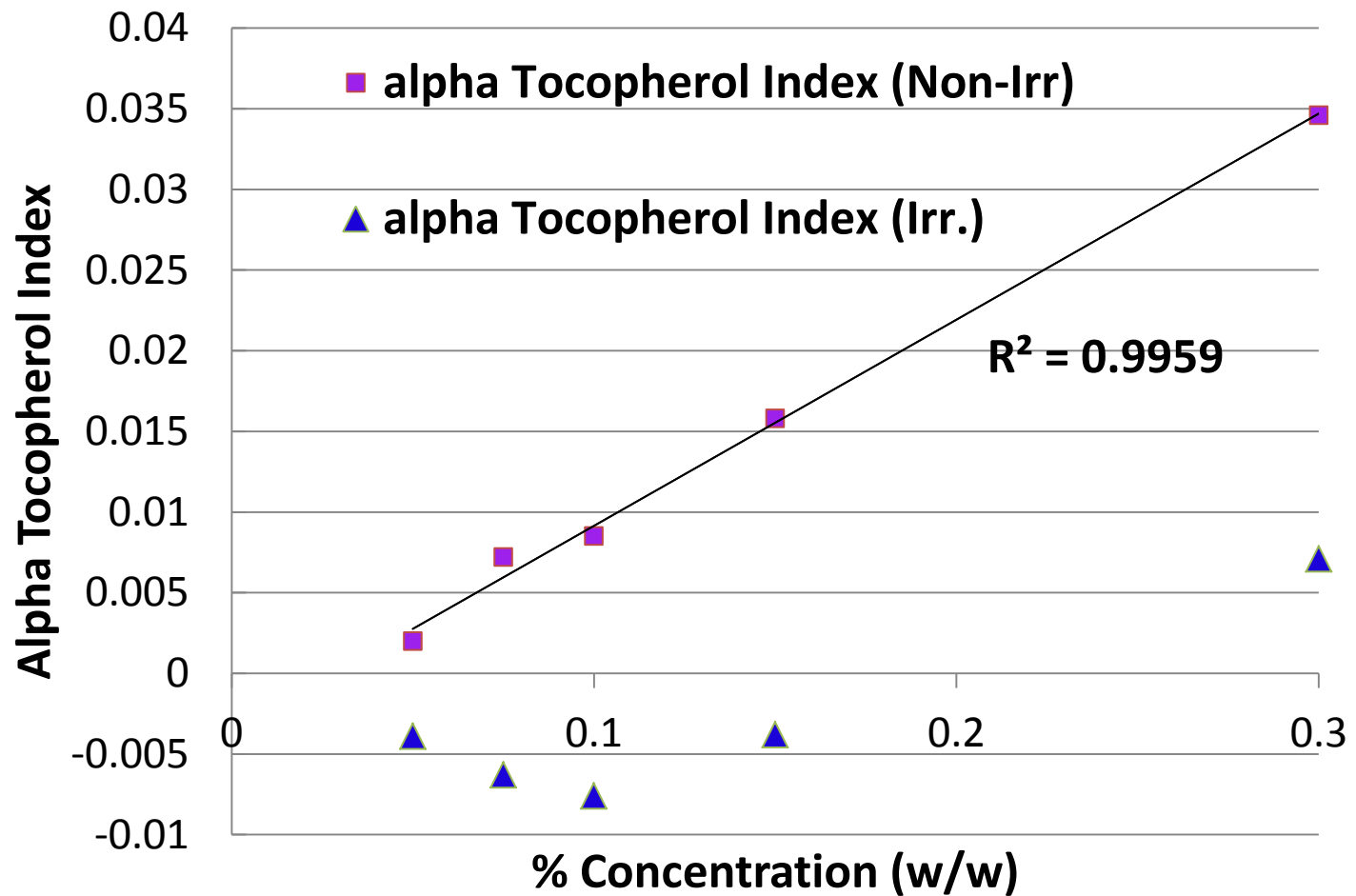
Test Methods

- **PBHP Index**: FTIR peak area between 1150 ~1125 cm^{-1} normalized using peak height at 1888 cm^{-1} .
- **α T Index**: FTIR peak area between 1275 ~1245 cm^{-1} normalized using peak between 1980 ~ 1850 cm^{-1} .
- **Oxidation Induction Time (OIT)**: Equilibrate at 200° C for 5 minutes under nitrogen and switch to oxygen at t=0 at a flow rate of 50 mL/min. OIT is measured when at time to an exotherm of 0.5 W/g.
- **Gravimetric Swell Ratio**: The swell ratios are measured in accordance with the procedure per ASTM D2765
- **Double Notched Izod Toughness**: Measurement is in accordance with procedure in ASTM F648

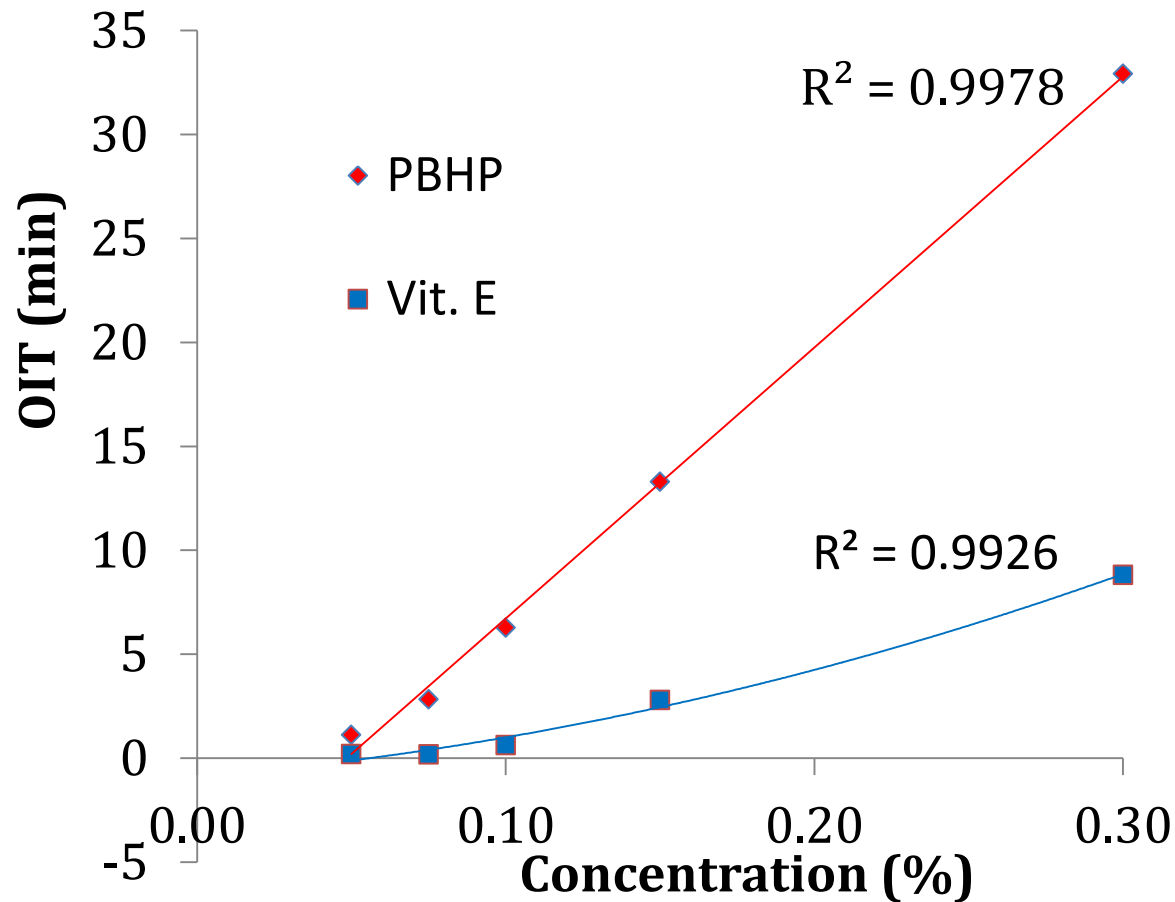
PBHP Index by FTIR



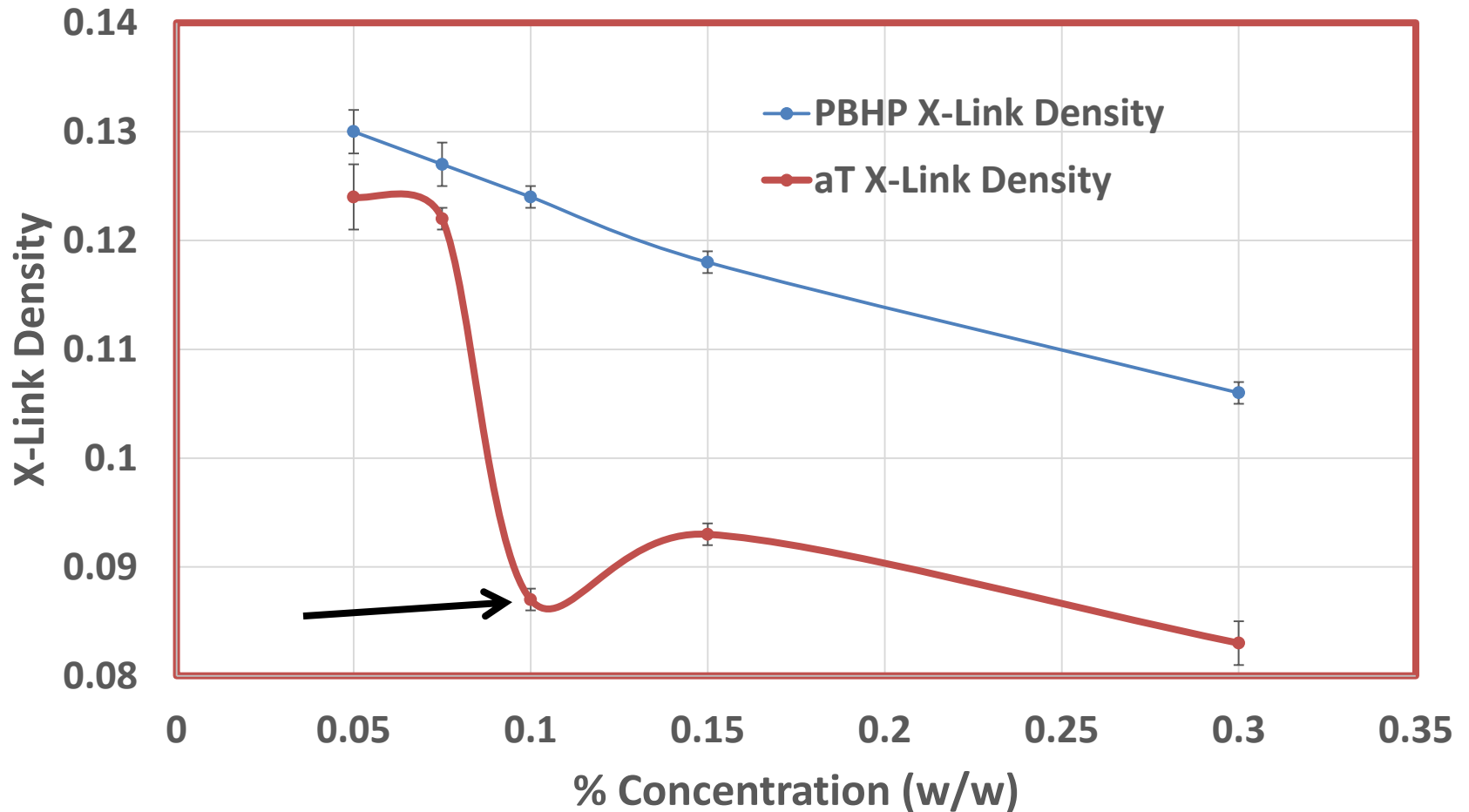
α T Index by FTIR



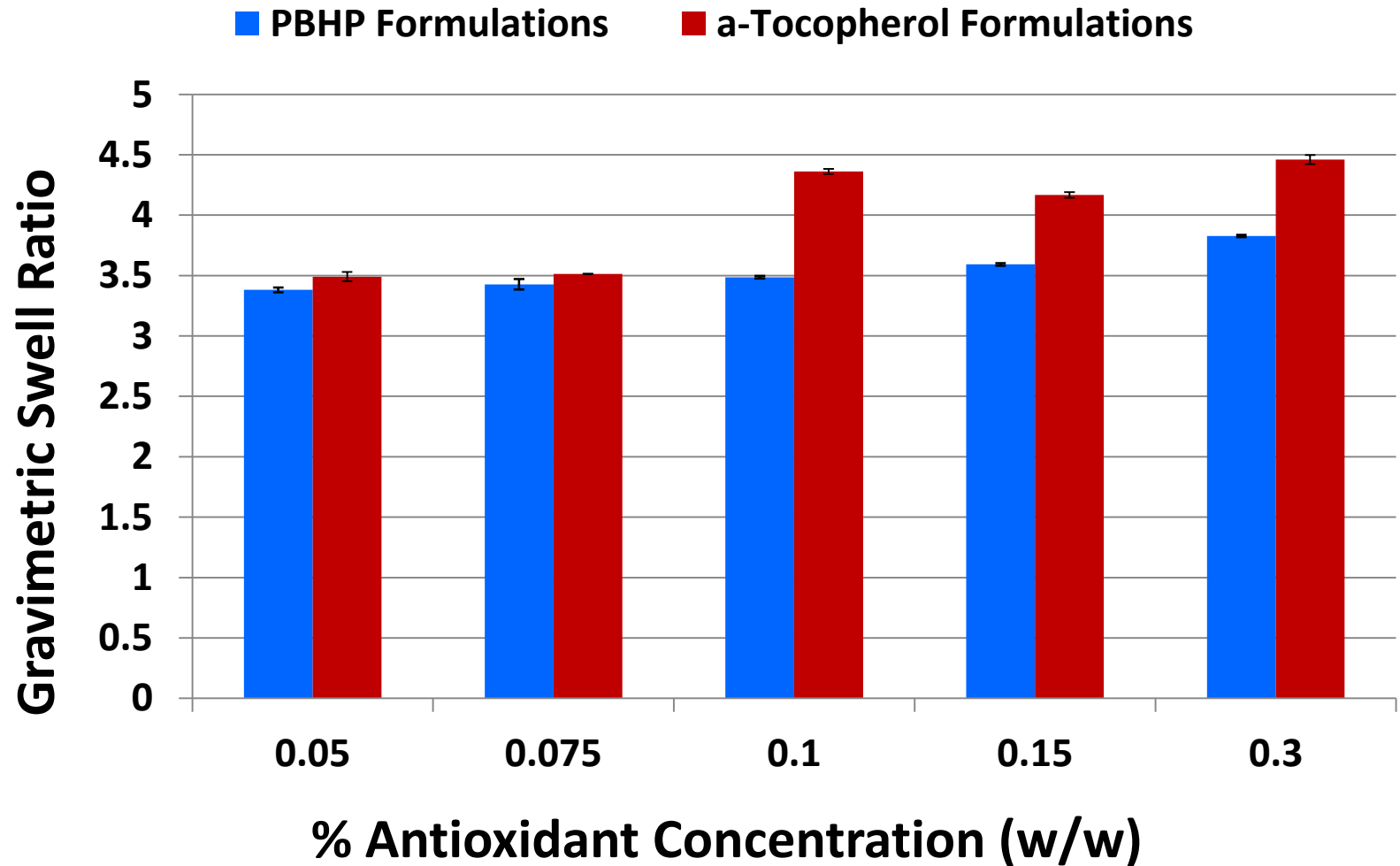
Oxidation Induction Time by DSC



X-Link Density by GSR



Swell Ratio by GSR



DNI Toughness

PBHP		α Tocopherol	
PBHP Sample ID	DNI Toughness (kJ/m ²)	α T Sample ID	DNI Toughness (kJ/m ²)
PBHP – 0.05%	71.2 \pm 2.2	α T – 0.05%	58.2 \pm 1.5
PBHP – 0.75%	72.5 \pm 0.6	α T – 0.75%	60.4 \pm 0.6
PBHP – 0.10%	72.9 \pm 2.0	α T – 0.10%	62.9 \pm 1.6
PBHP – 0.15%	76.0 \pm 1.3	α T – 0.15%	65.3 \pm 2.5
PBHP – 0.30%	79.7 \pm 2.0	α T – 0.30%	75.4 \pm 1.0

Next Steps

- Evaluate and report other properties of the formulations such as tensile, fatigue and creep resistance
- Compare the properties after artificial aging for 2, 4, 6, and 10 weeks using the ASTM F2003 protocol
- Representative wear testing head-to-head of representative formulations in a wear simulator.

Conclusions

- Successful characterization of the antioxidant formulations has been demonstrated by FTIR and DSC.
- The depletion in concentration upon irradiation has been verified by FTIR.
- Good linearity of response has been demonstrated as a function of concentration for both antioxidant materials by OIT and FTIR, particularly for **PBHP** formulations.
- The results confirm a more rapid depletion of the measurement signal for the α T formulations relative to that for the **PBHP** formulations upon irradiation by FTIR.
- This has been verified at each concentration by comparison of OIT measurements suggesting a relatively higher rate of depletion of the α T formulations.
- **PBHP** formulations demonstrate better crosslinking efficiency relative to the corresponding α T formulations at the same concentration upon irradiation to the same dose.
- Irradiated formulations based on **PBHP** show higher impact toughness than the corresponding α T formulations at lower concentrations.