Fiber-matrix Adhesion and Thermal Stability of HDPE/GF Composites

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High-Temperature Pressure Vessels

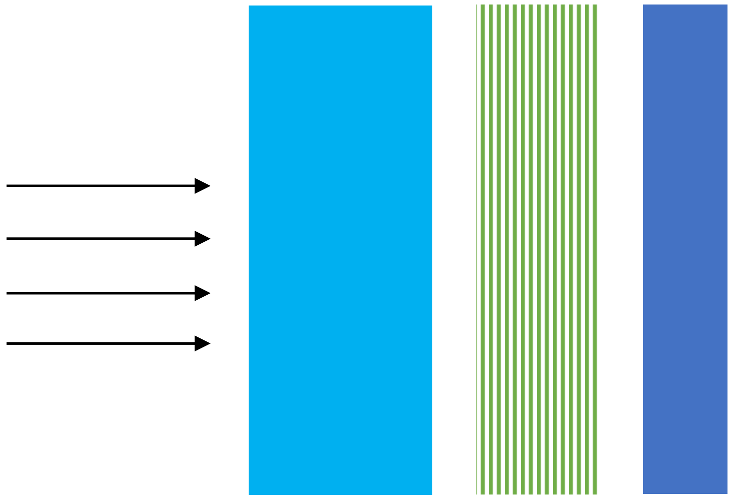
- Desire to replace metallic components with fully polymeric and composite structures

- Multi-layered structure:

    1. Chemical resistant inner layer, protection from carrier fluids
    2. Reinforcement layer providing structural integrity, at high temperatures and pressures
    3. Outer jacket to protect from environmental conditions (moisture, UV, impact)
    4. Reduce/eliminate permeation of carrier fluids to the environment

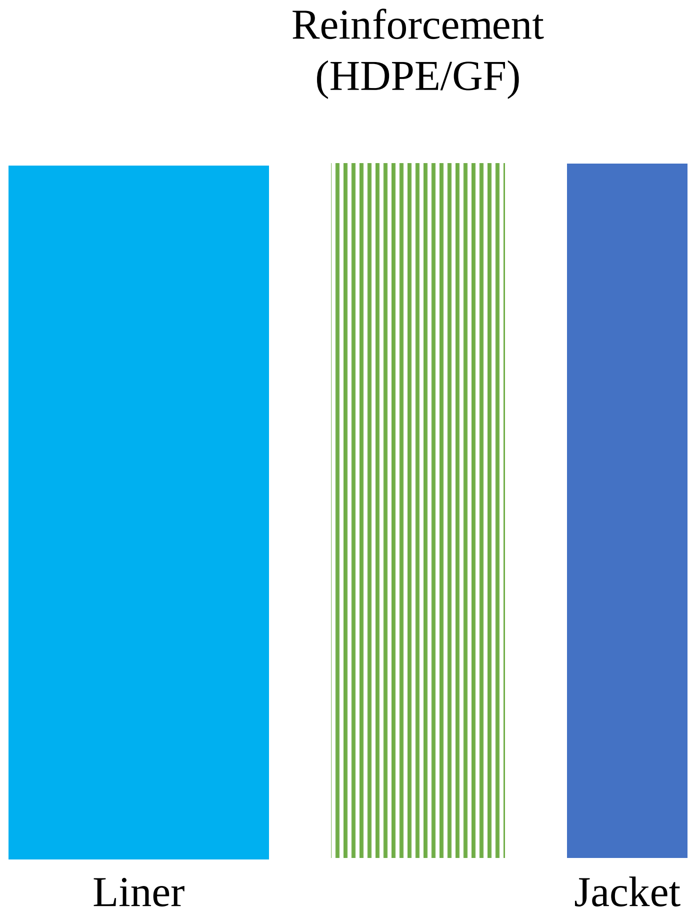
https://cdn.shawcor.com/hg/medialibraries/shawcor/corporate/pdfs/cps/flexflow.pdf

Transport Fluids
- Hydrocarbons
- Water
- Brine
- Methanol
- Hydrogen



High-Temperature Pressure Vessels

- Bonded or non-bonded (dry glass) reinforcement
- Increasing temperature and pressure capabilities requires retention of dimensional stability
- Consider reinforced liners and jacket layers
- Resistance to rapid gas decompression (liner)
- Permeation and moisture resistance (liner / jacket)
- Thermo-oxidative resistance (jacket / reinforcement in non-jacketed products)
- Benefits from accelerated testing for material screening



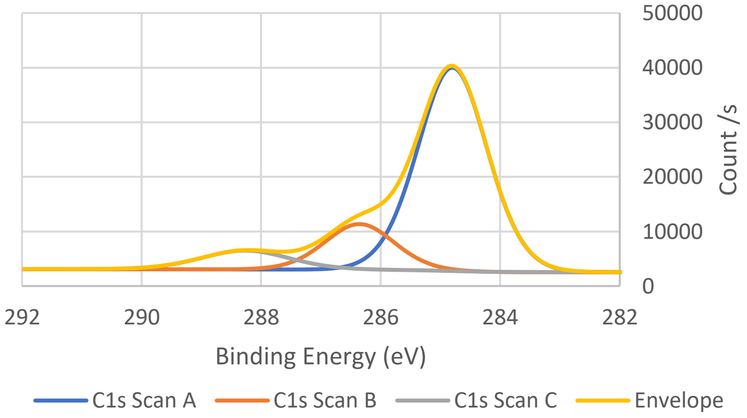
Materials

Formulations and Nomenclature

Compound	Coupling Agent, PE- MAH (wt%)	Stabilizer, ST (wt%)	Glass Fiber (wt%)
Neat	0	0	0
3VA	0	3	0
3MA	3	0	0
3MA 3ST	2.91	3	0
8MA	8	0	0
8MA 3ST	7.76	3	0
12.5MA	12.5	0	0
12.5MA 3ST	12.125	3	0
3MA 30GF	*2.1*	*0*	*30*
12.5MA 3ST 30GF	*8.487*	*2.1*	*30*

- High density polyethylene (HDPE) matrix
  - Low cost, facile low-temperature processing, low moisture absorption
  - Poor adhesive properties
- Maleic anhydride grafted HDPE coupling agent (MA)
  - Promotes adhesion to reinforcing fibers
  - Concern with long-term stability at high temperature
- Zinc-based antioxidant (ST)
- E-Glass fiber
  - Proprietary silane sizing for PE matrices

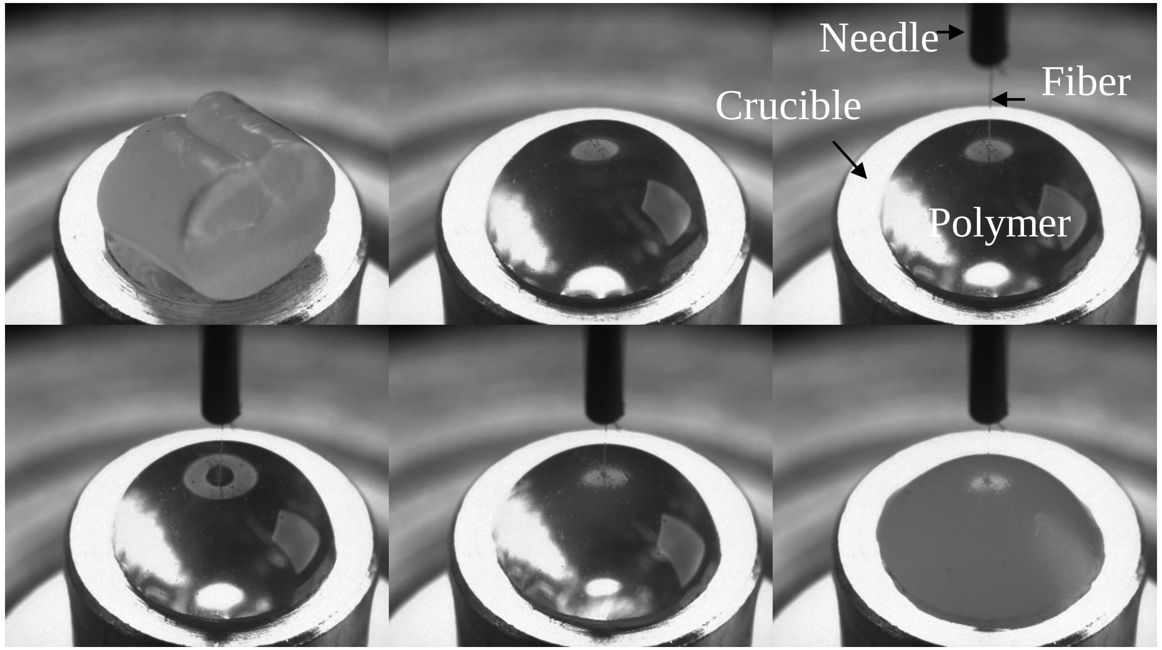
Element	Atomic %
C	53.25
N	2.55
O	30.02
Si	11.78

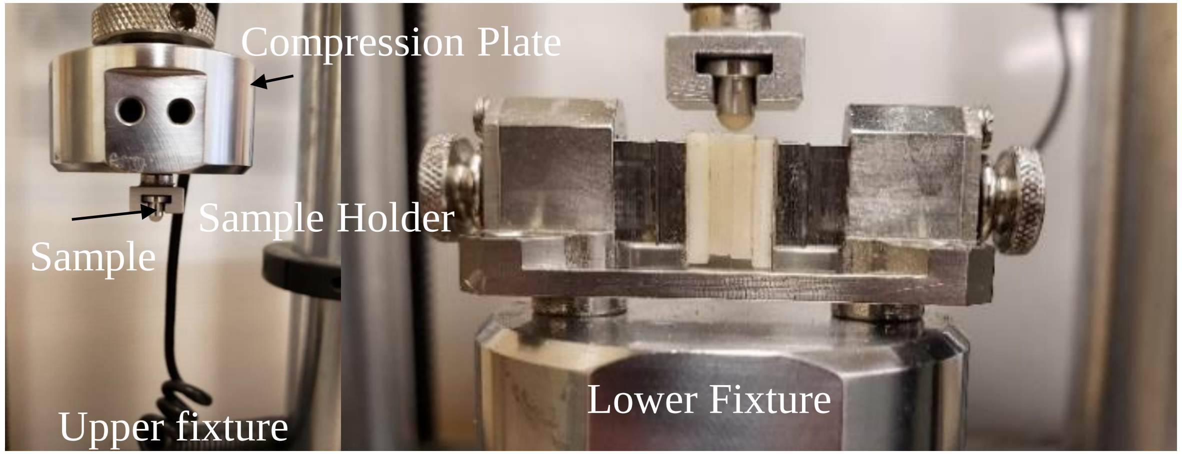


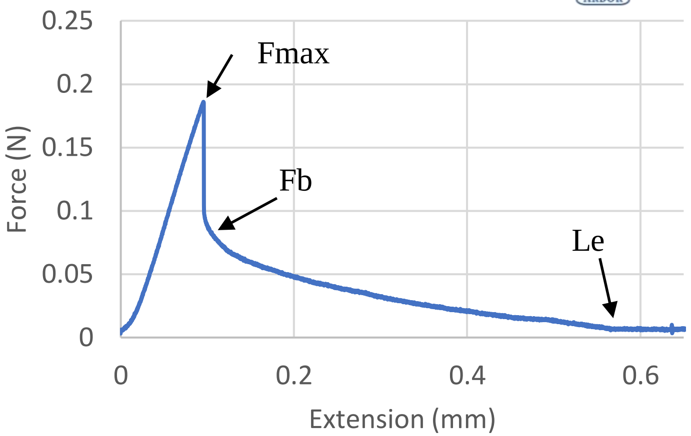
Name	Peak BE	Atomic %
C1s Scan A	284.8	74.56
C1s Scan B	286.36	16.78
C1s Scan C	288.27	8.66

Methodologies

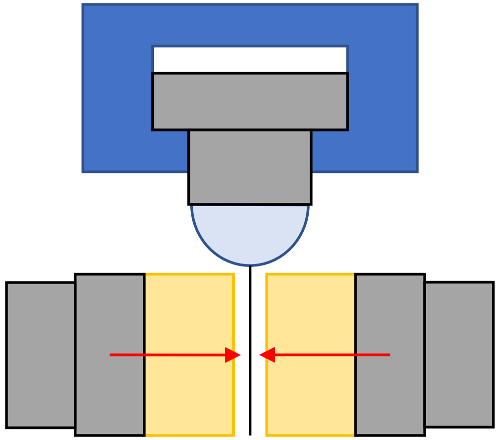
Single-fiber pullout



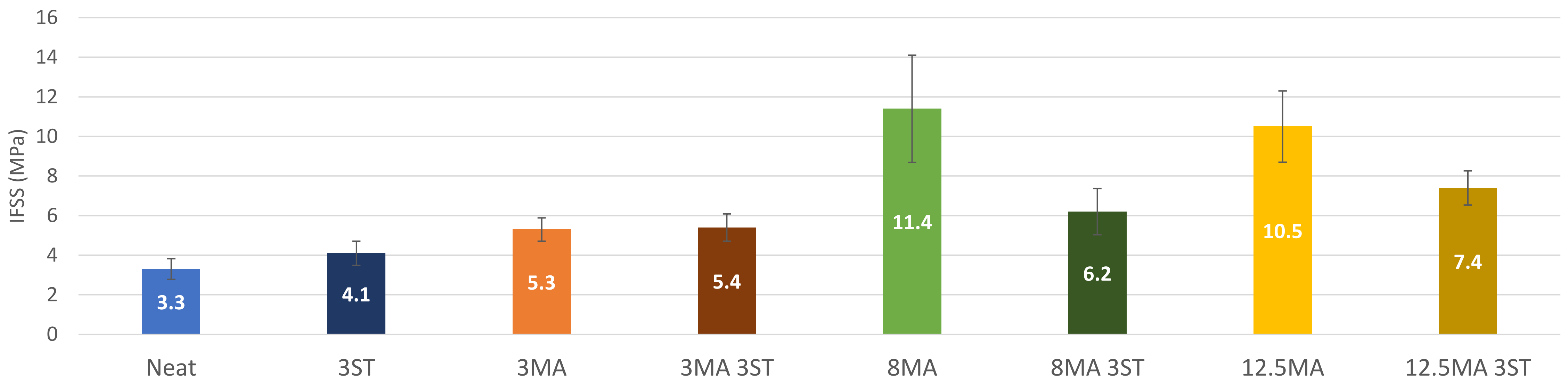


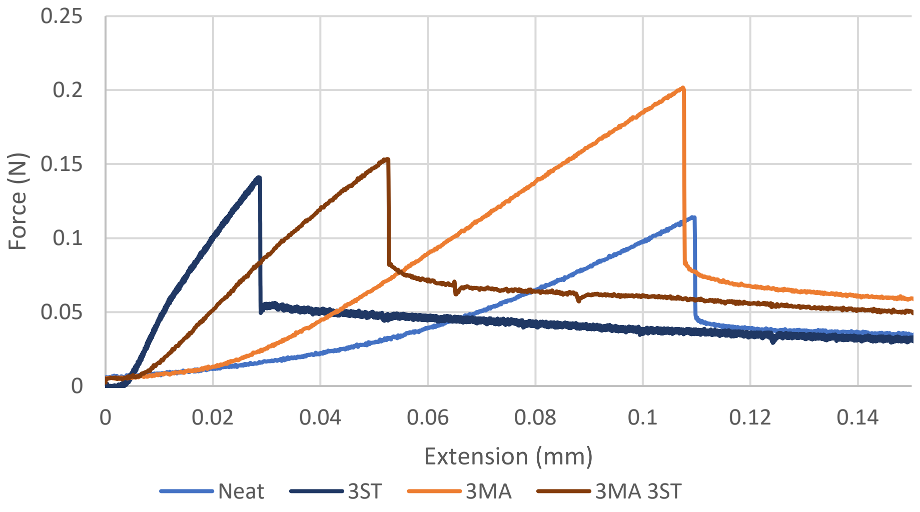


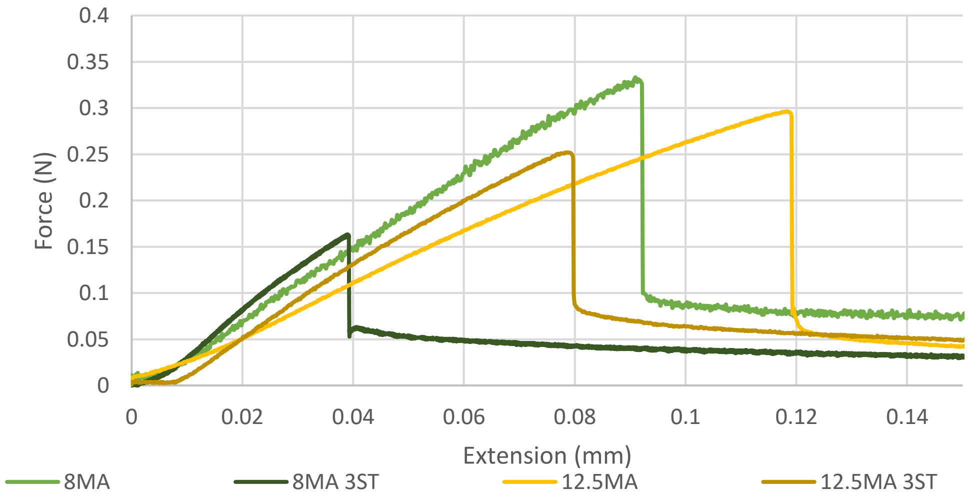
$$IFSS = \frac{Fmax}{\pi D L_e}$$



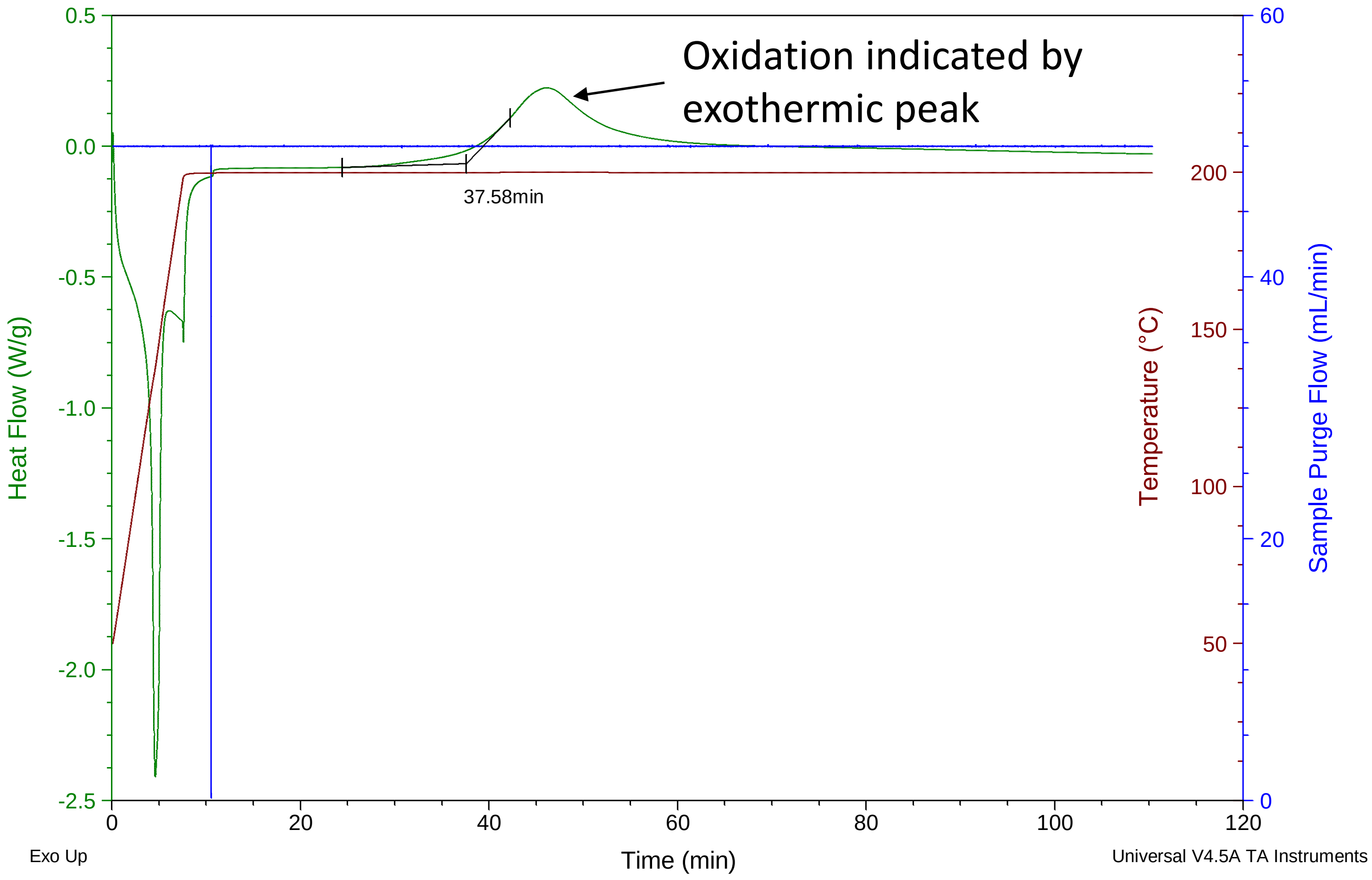
Apparent Interfacial Shear Strength







Methodologies
Oxidation induction time

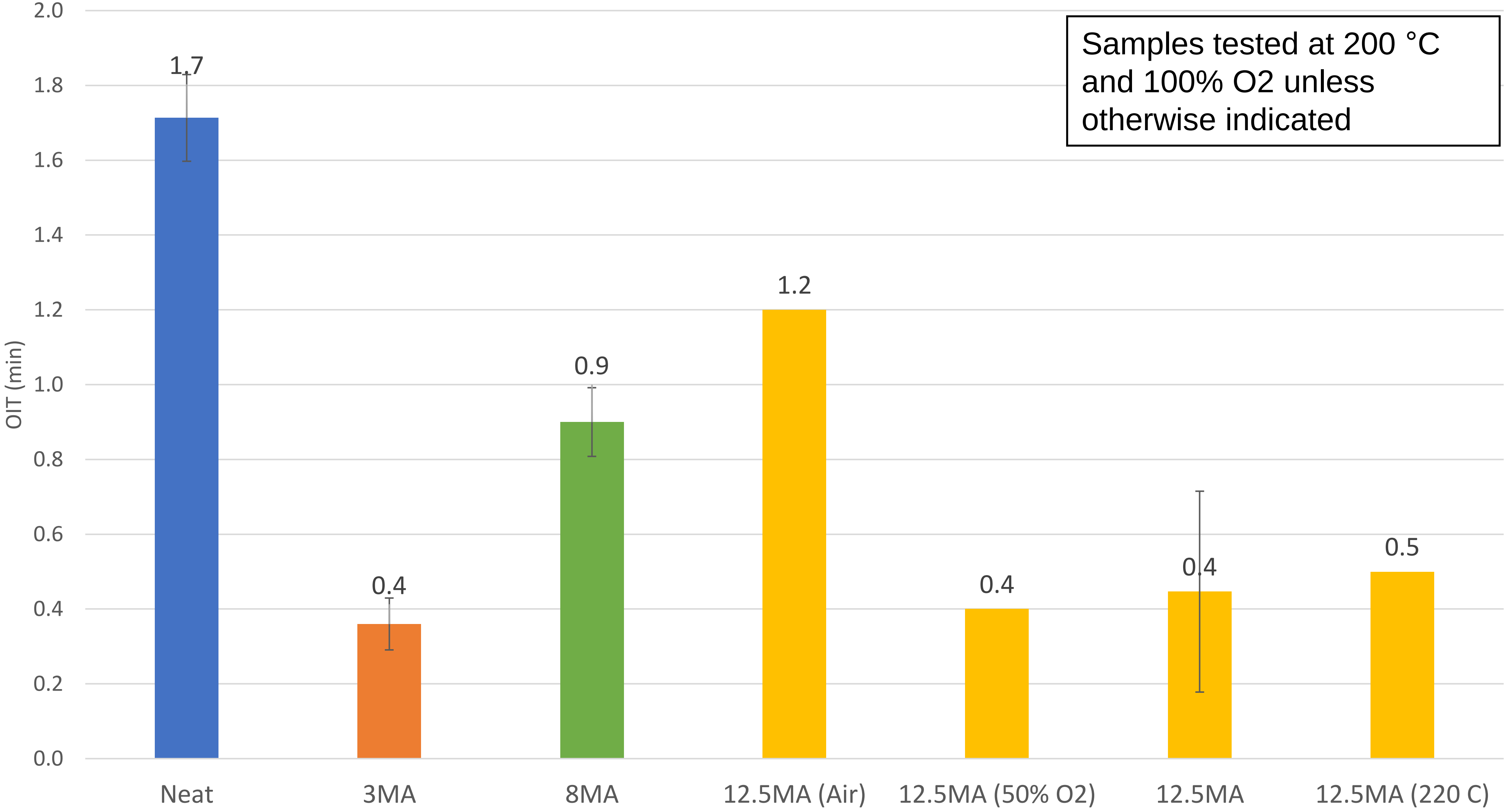


Test is conducted using DSC

- Samples cut to size of 1 x 2 x 3 mm and placed in open crucible
- Heated in a nitrogen atmosphere to 200 °C and held isothermal for 3 min
- Gas is then switched to pure oxygen and oxidation is indicated by an exotherm
- OIT is measured by the onset of this exotherm subtracted by the total time under nitrogen

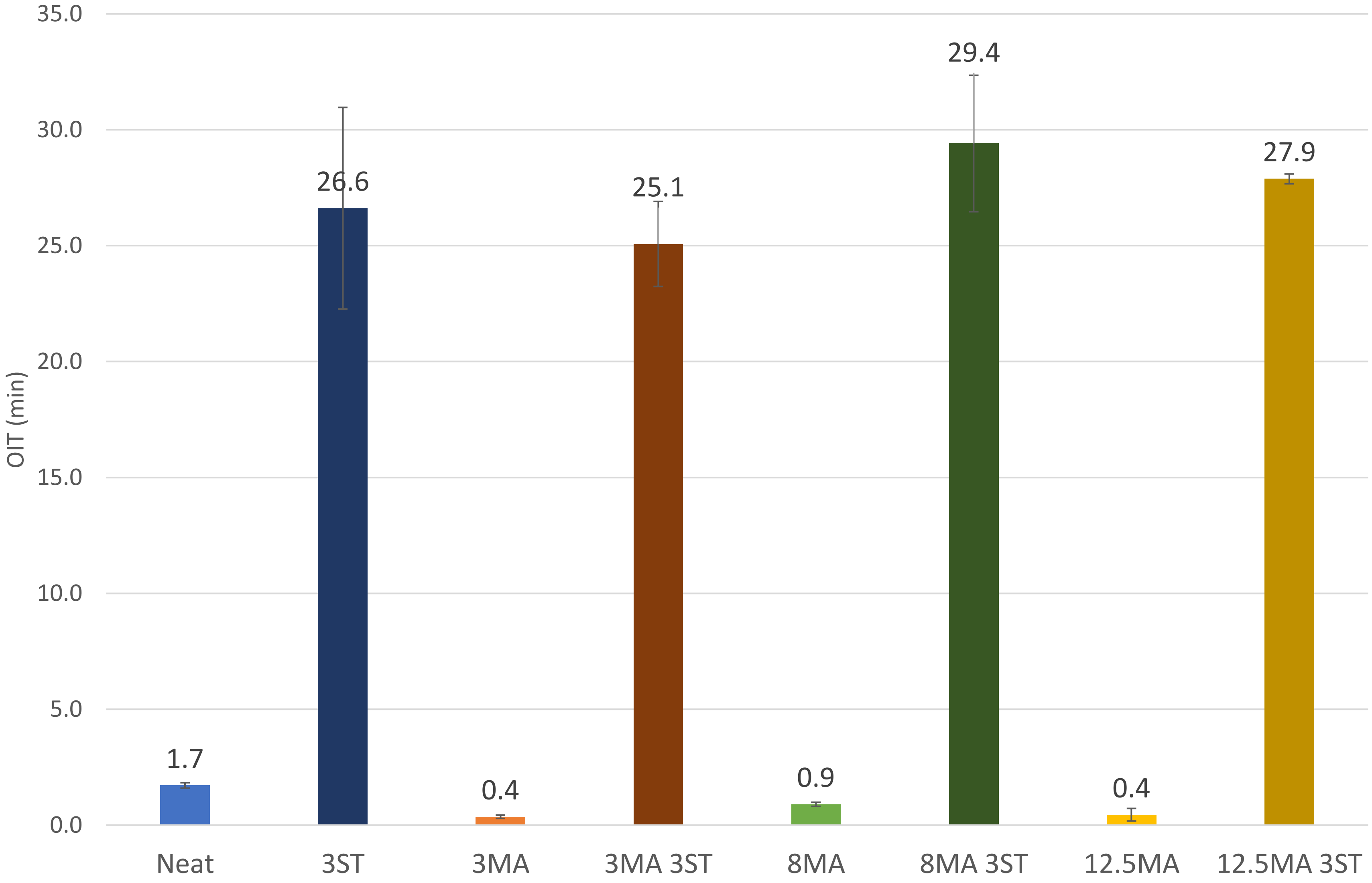
Oxidation Induction Time (OIT)

Effect of Coupling Agent



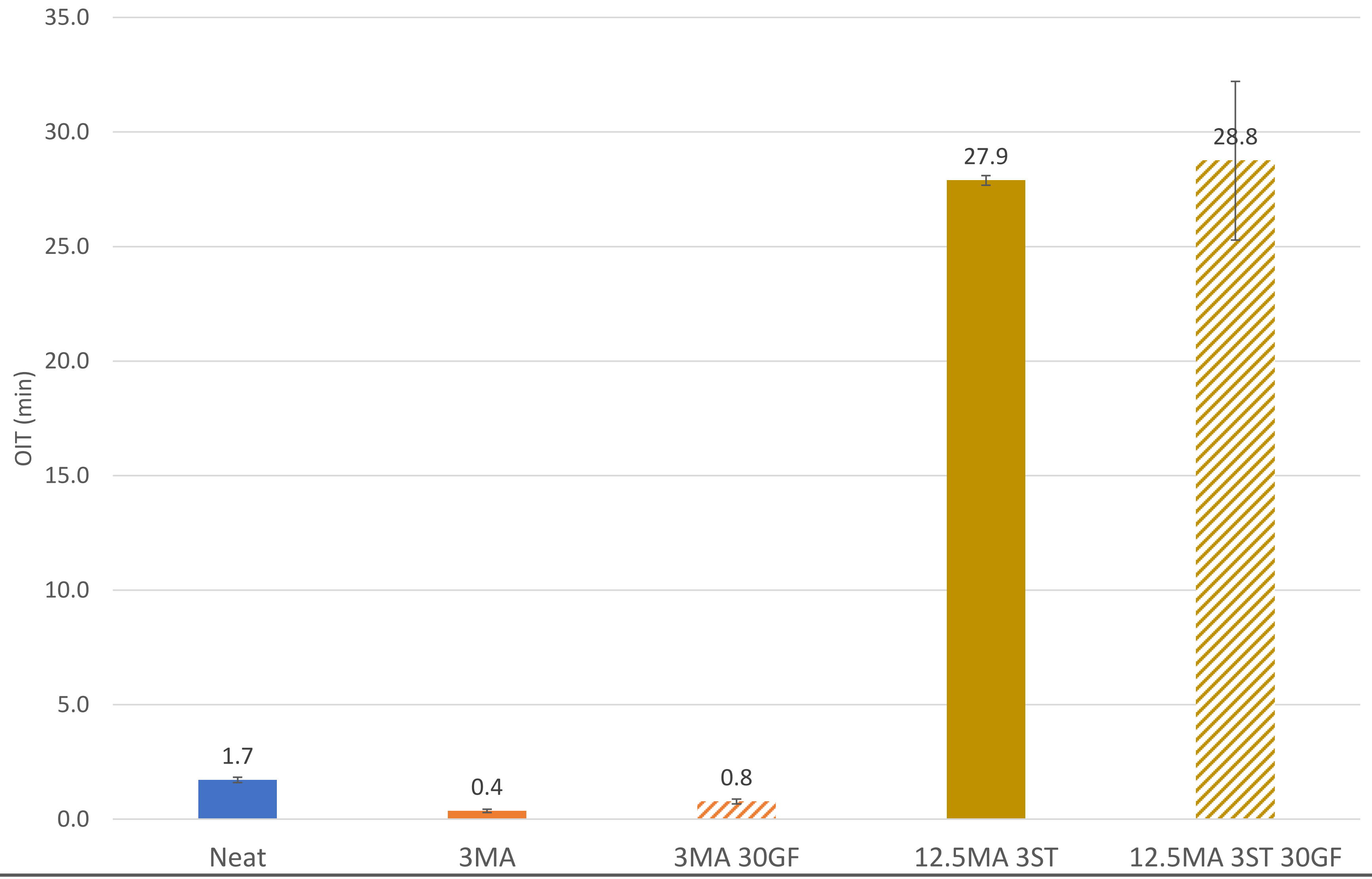
Oxidation Induction Time (OIT)

Effect of Stabilizer



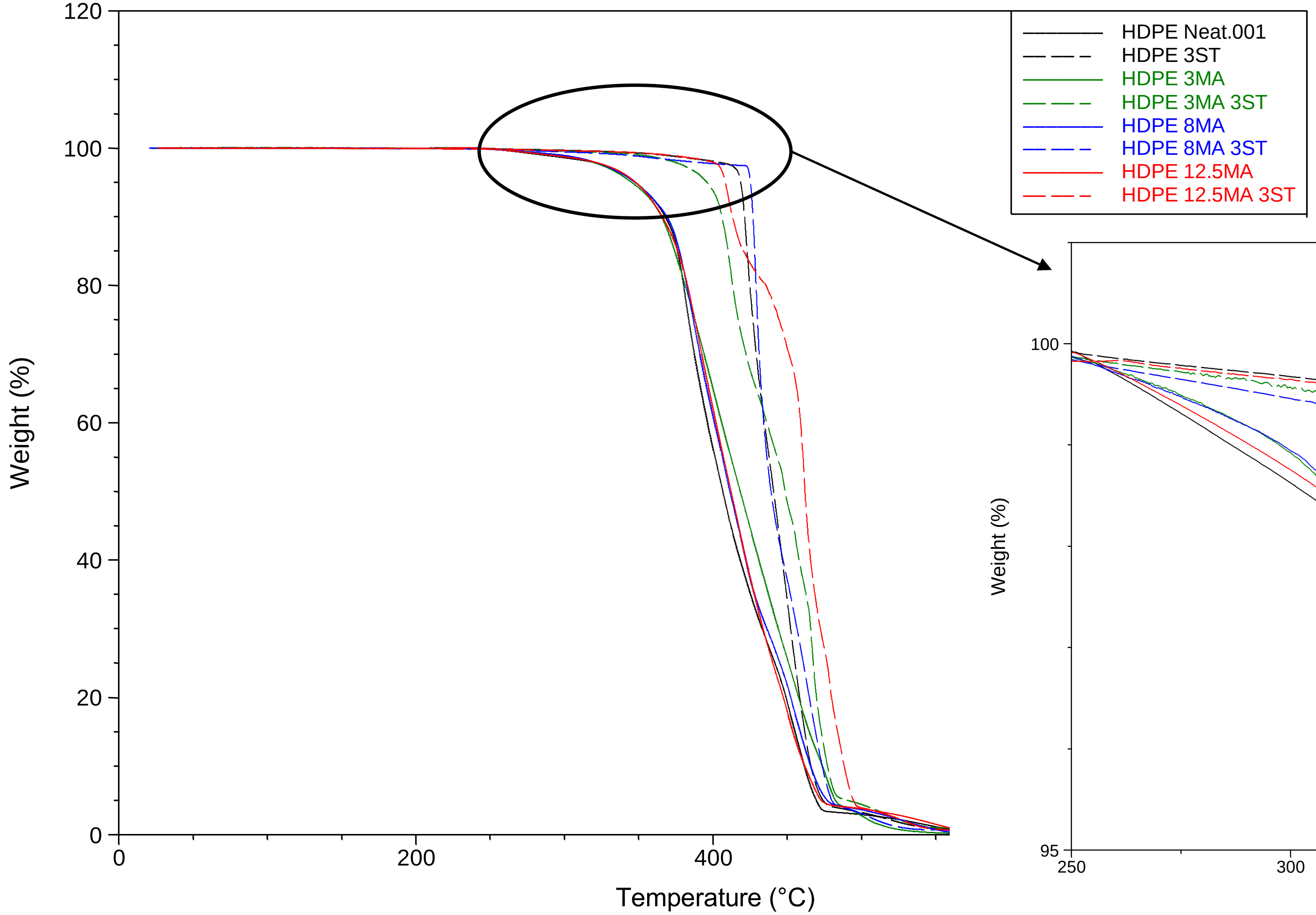
Oxidation Induction Time (OIT)

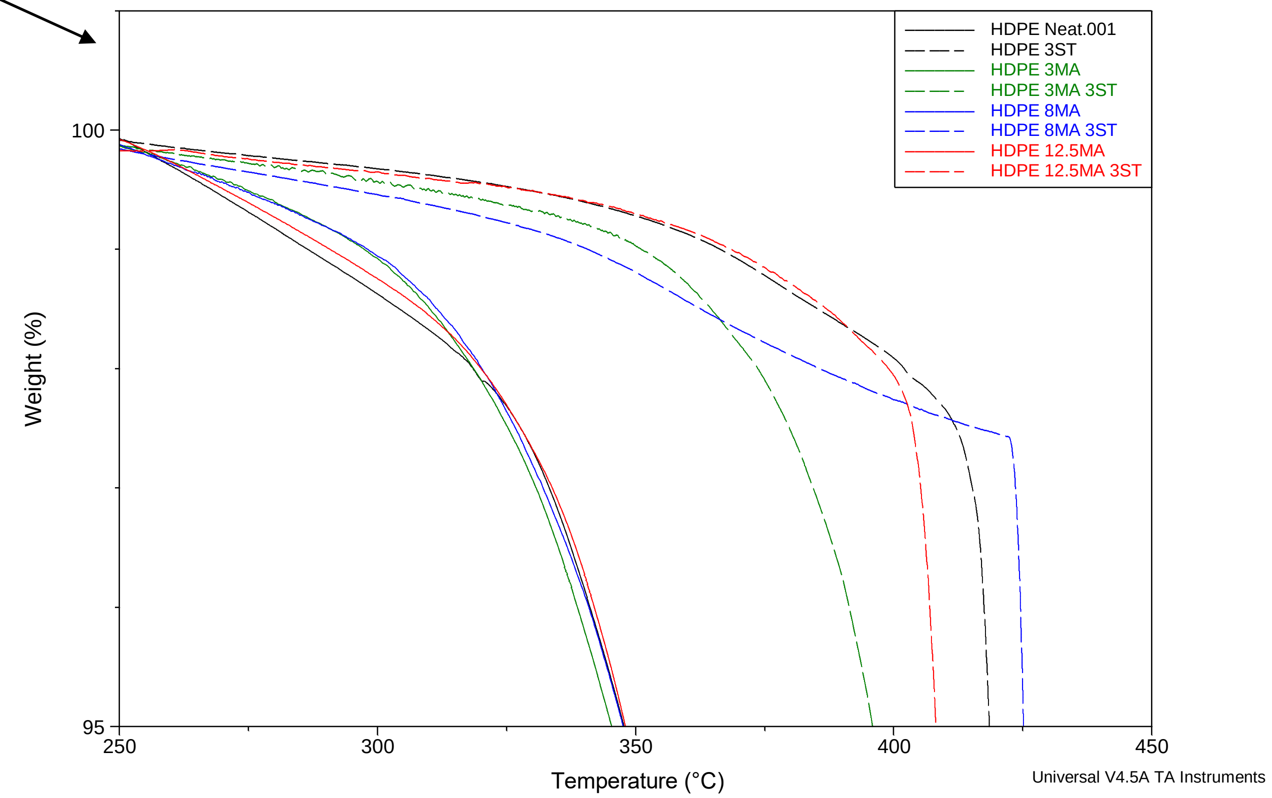
Effect of Glass Fiber



Thermogravimetric Analysis

10 °C/min Scan





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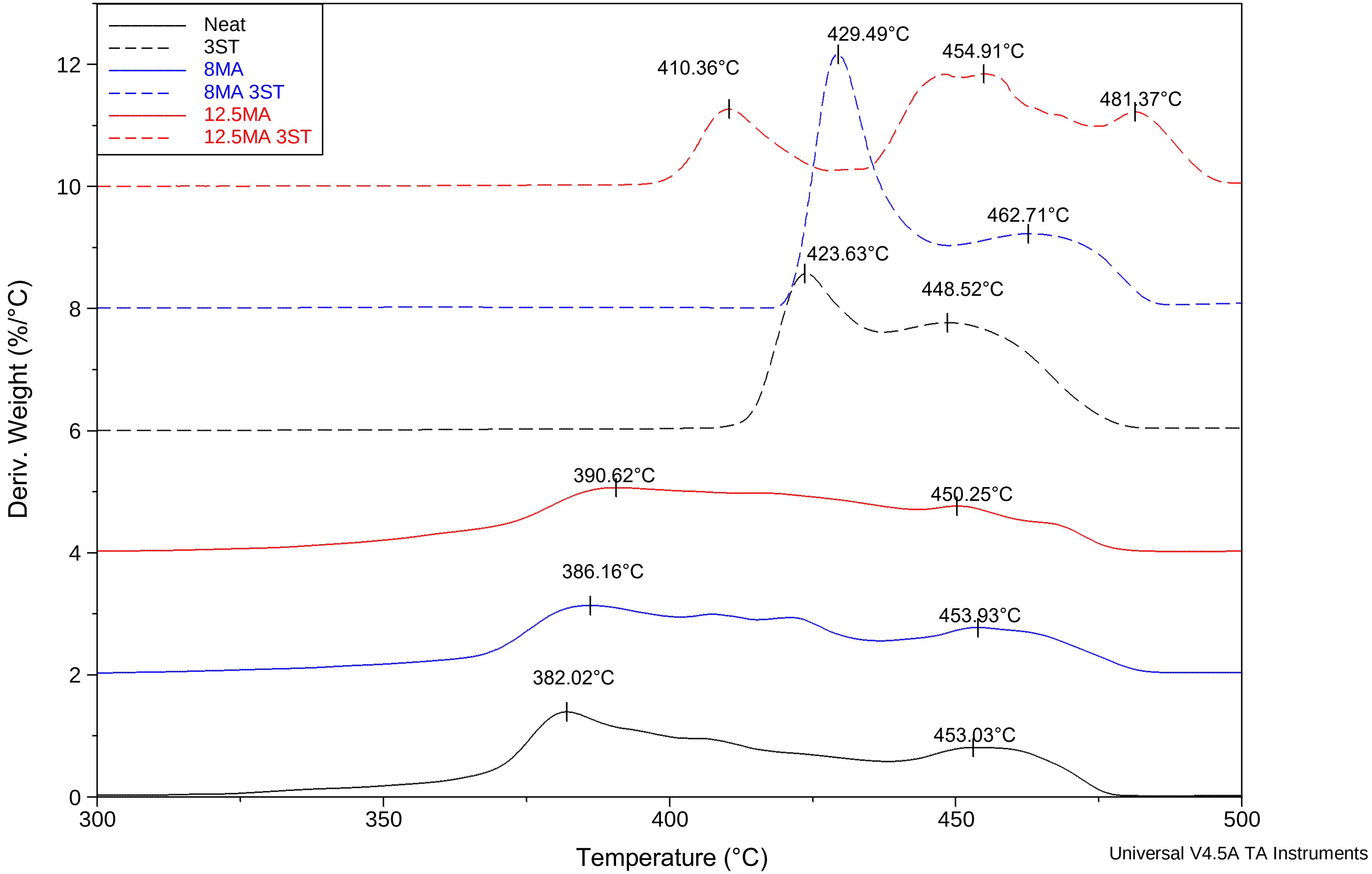
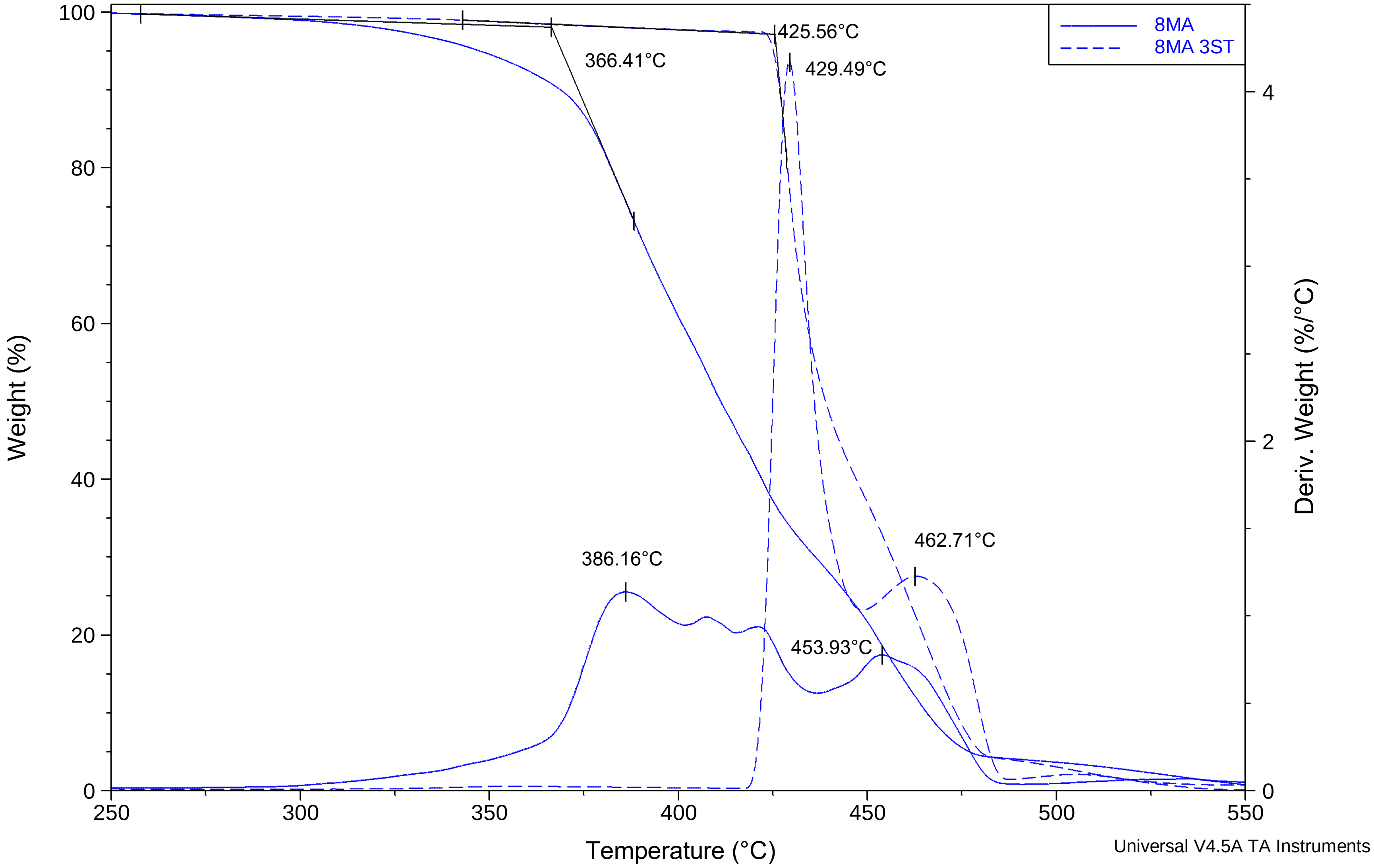


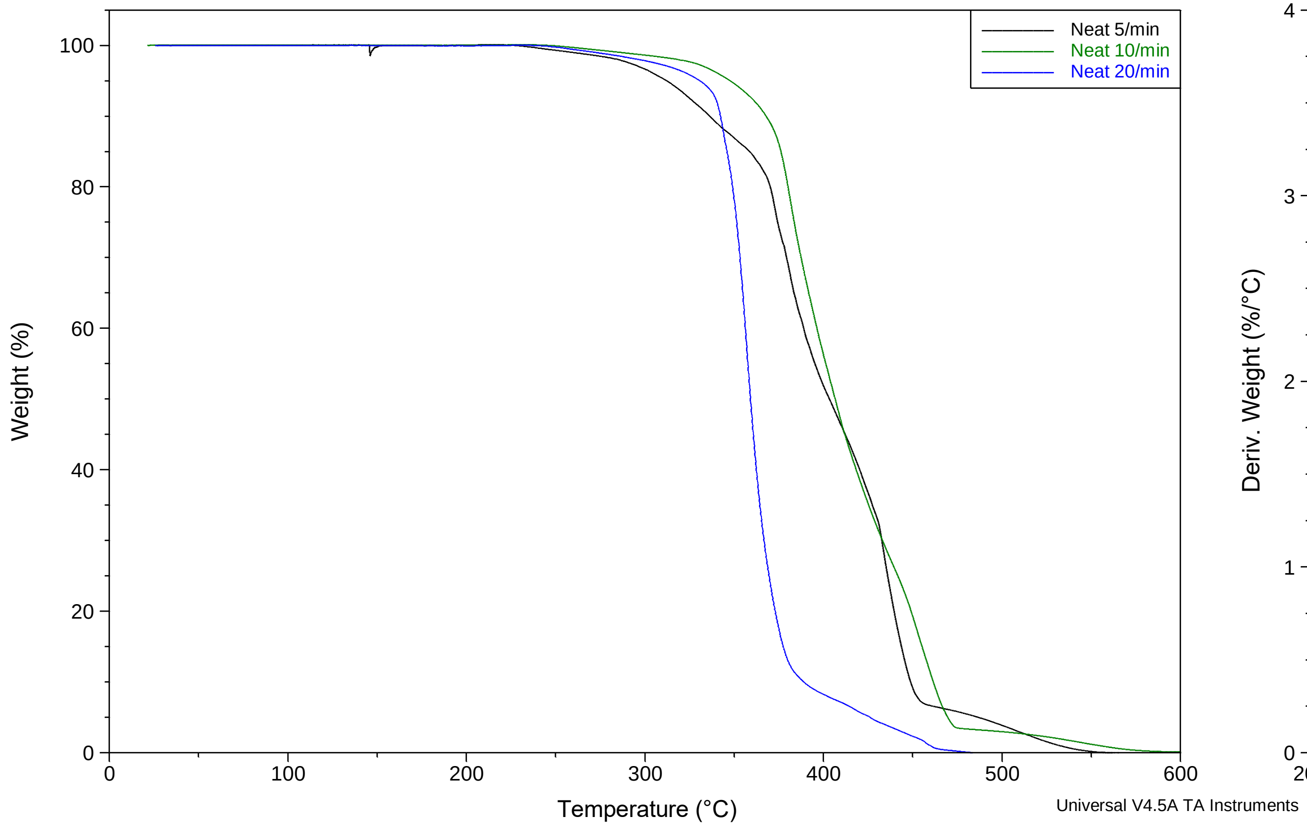
Thermogravimetric Analysis

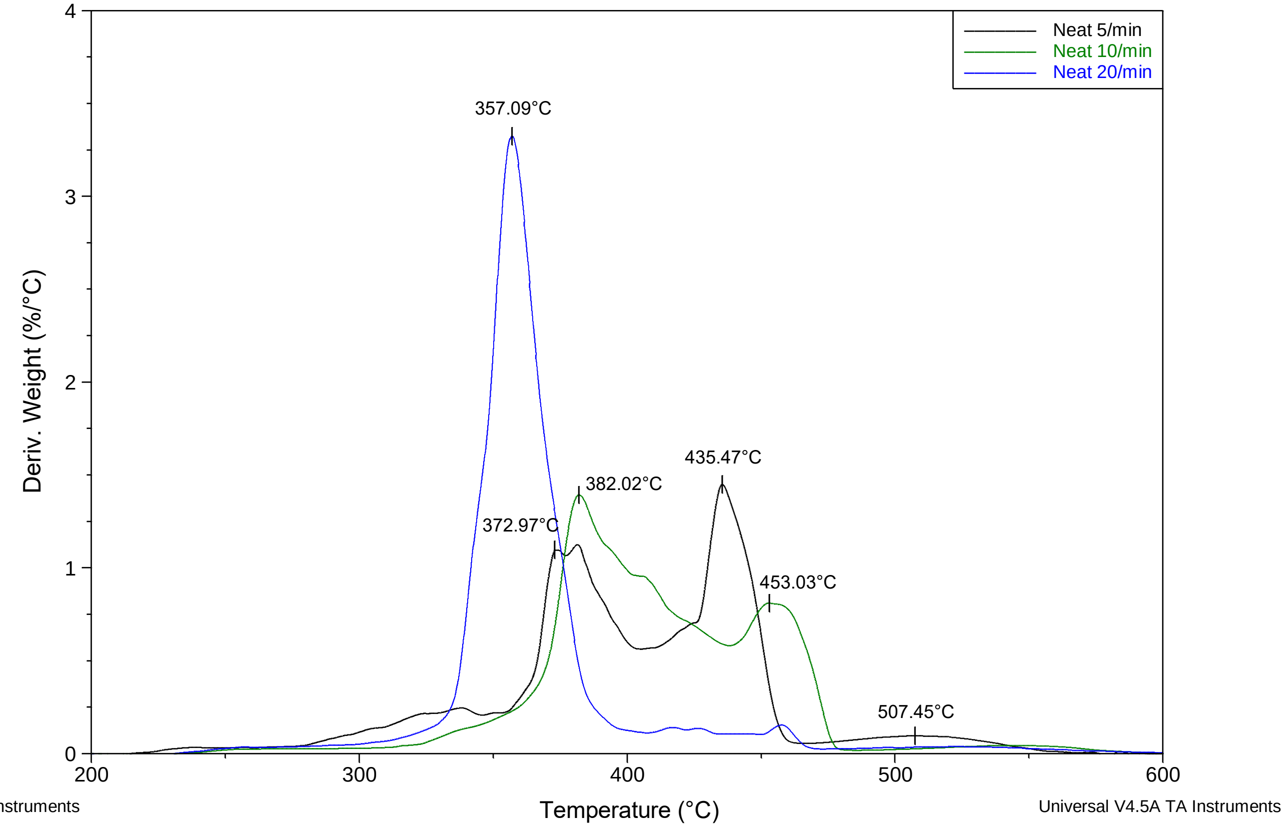
HDPE with 8MA and Stabilizer



Thermogravimetric Analysis

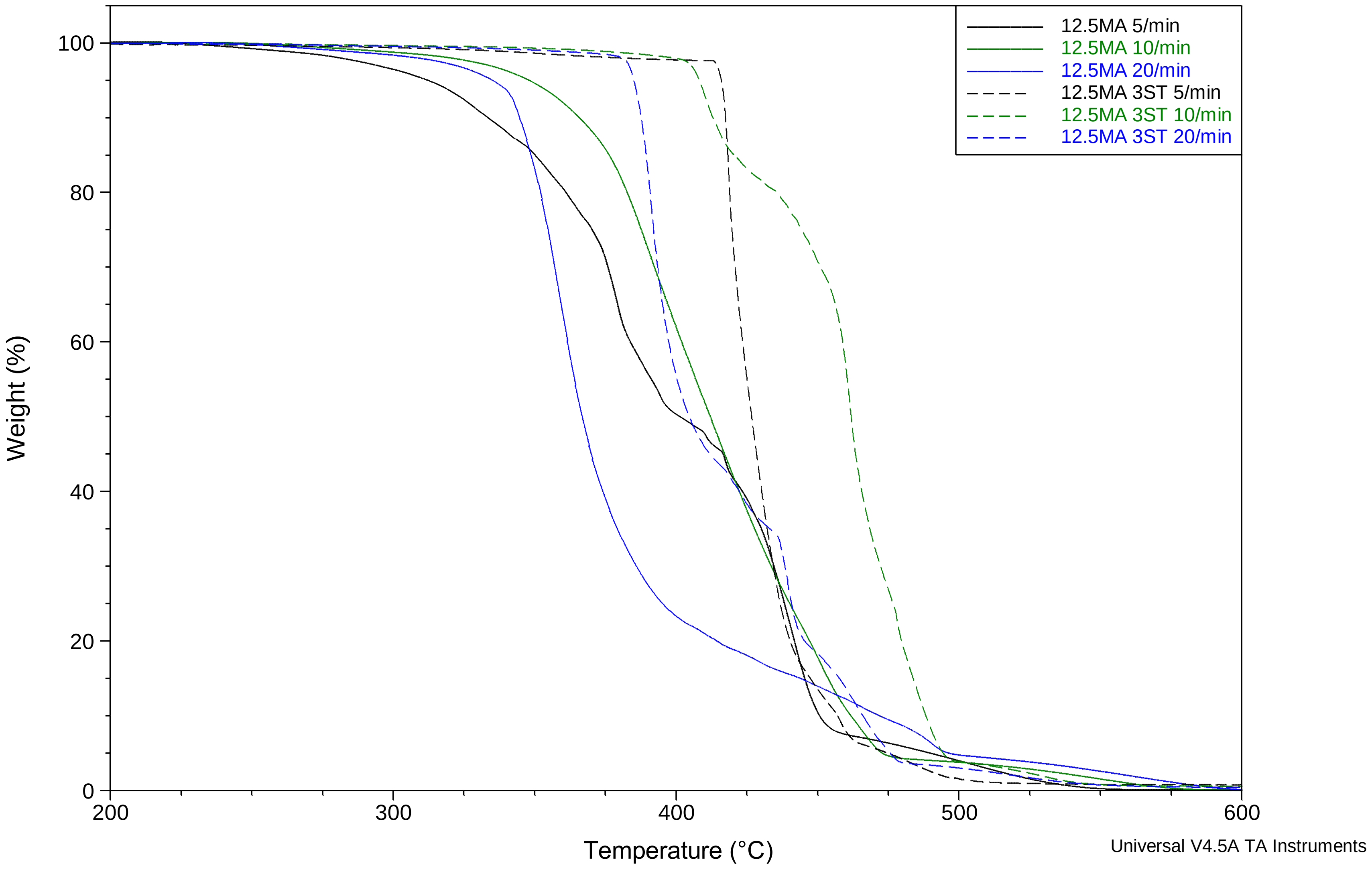
Effect of Heating Rates: Neat HDPE





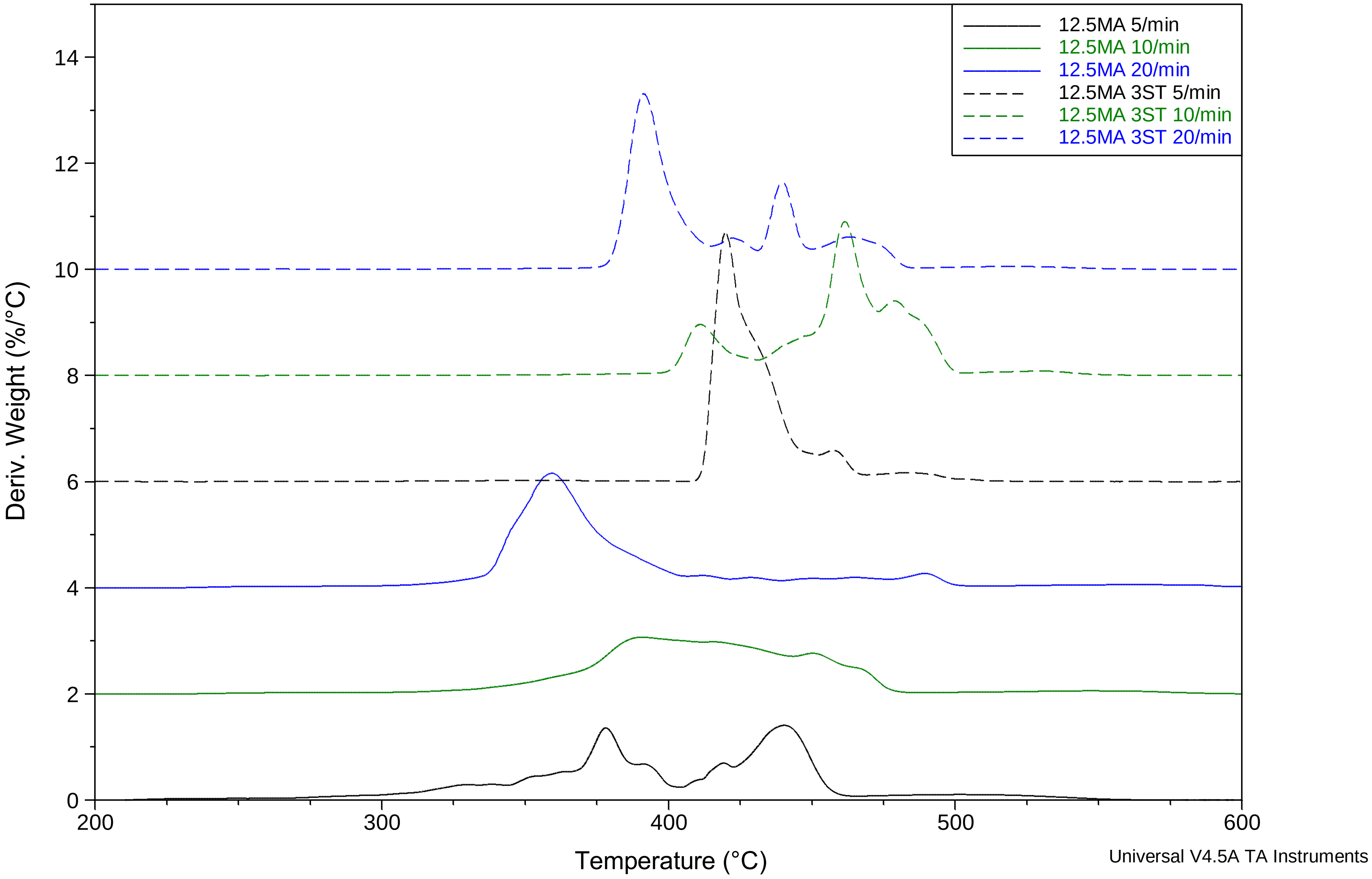
Thermogravimetric Analysis

Effect of Heating Rates: HDPE with 12.5MA and Stabilizer



Thermogravimetric Analysis

Effect of Heating Rates: HDPE with 12.5MA and Stabilizer



Contact Angle and Surface Energy

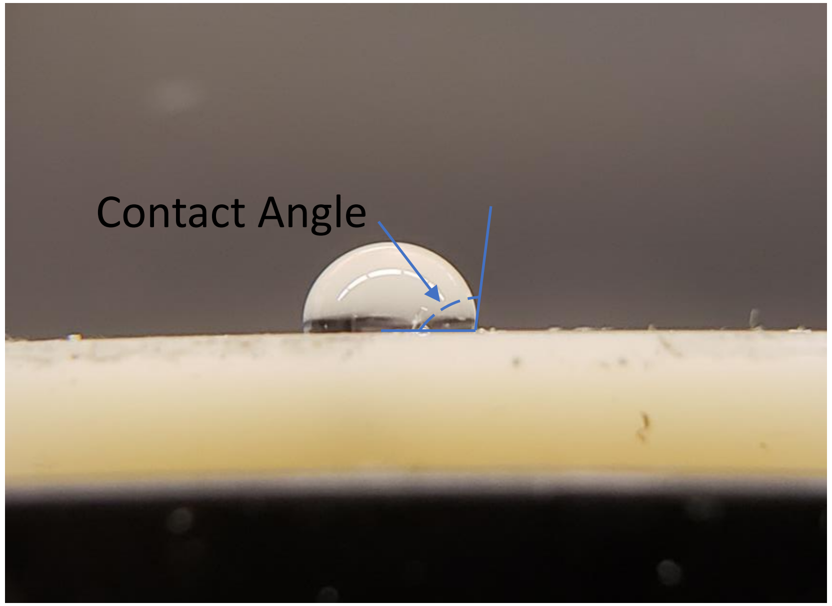
- Functionality of the HDPE compounds determined by surface energy characterization using Fowkes method

- Contact angles were measured between HDPE and diiodomethane, completely dispersive, and deionized water, polar and dispersive

- Two component surface energy characterization is given by the Owens-Wendt model. Total surface energy is the sum of the polar and dispersive components

$$\left(\gamma_s^D \gamma_l^D\right)^{1/2} + \left(\gamma_s^P \gamma_l^P\right)^{1/2} = \gamma_l\,(cos\theta + 1)/2$$

Sample	Theta (diiodomethane)	Theta (Water)	γ (dispersive)	γ (polar)	Surface Energy (mJ/m²)
HDPE Neat	54.04	95.67	32.00	4.98	*37*
HDPE 3ST	53.24	97.55	32.45	3.08	*36*
HDPE 3MA	52.16	95.31	33.06	4.71	38
HDPE 3MA 3ST	51.26	95.72	33.57	4.05	38
HDPE 8MA	49.29	91.95	34.67	6.92	**42**
HDPE 8MA 3ST	49.93	95.03	34.32	4.25	39
HDPE 12.5MA	49.59	91.46	34.50	7.49	**42**
HDPE 12.5MA 3ST	48.99	94.88	34.84	4.09	39



Summary

- Without stabilizer, IFSS increases up to 8% maleic anhydride with no changes observed up to 12.5%
- The thermal stabilizer dramatically reduces IFSS for these highly compatibilized materials by inhibiting the functional groups
- Oxidation occurs instantaneously for all compounds with maleic anhydride but is greatly delayed by the stabilizer
- Oxidation induction time does not depend on the compatibilizer content with or without the stabilizer
- Thermal stability measured by TGA is comparable to results obtained by OIT, but some differences with MA content were observed
- Thermal decomposition kinetics of compounds changes with varying heating rates