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Improved Processing of Highly Filled Calcium Carbonate Compounds

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***Presenter**

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ABSTRACT

Calcium carbonate treated with an interfacial agent such as stearic acid or a stearate is often used in place of untreated calcium carbonate to take advantage of improved lubricity and wetting characteristics. These characteristics typically lead to lower mixing viscosity, improved filler dispersion and better flow properties. In this paper, the effect of fatty acid derivatives on polypropylene compounds highly filled with calcium carbonate, both treated and untreated, will be discussed. The use of fatty acid derivatives can be effective for lowering extrusion pressure leading to throughput increases. The overall effect on filler addition, viscosity, mixing and processing properties will be shown for one class of fatty acid derivatives.

EFFECT MECHANISMS OF THE ADDITIVES:

- Tribological
  - Adhesives
  - Lubricants
  - Surfactants
- Molecular

EFFECT MECHANISMS OF THE ADDITIVES:

- Tribological
  - Adhesives
    - Increased interfacial forces created by surface attachment
    - Increase energy required to break adhesive bonds causing increased shear
  - Lubricants
    - Function to minimize the frictional forces between moving surfaces
    - Can be divided into internal and external
    - Internal is polymer:polymer, polymer:filler interaction
    - External is polymer:hot metal, filler:hot metal interaction
  - Surfactants
    - Create a surface active film via polar and non-polar ends
    - Polar end absorbs/bonds to a surface
    - Wetting of the filler allows for improved low energy dispersion
    - Similar to lubricants effect

EFFECT MECHANISMS OF THE ADDITIVES:

- Molecular
  - Chemical alteration of molecular weight average and number
  - In polypropylene, addition of peroxide results in chain scission
  - Lower molecular weight results in increased flow rates/low viscosity

TYPICAL PROPERTIES OF HIGHLY FILLED COMPOUNDS:

- Increased:
  - ✓ Viscosity *(-Negative)*
  - ✓ Flexural modulus *(+Positive)*
  - ✓ Heat deflection temperature (HDT) *(+Positive)*
  - ✓ Dimensional stability *(+Positive)*

- Decreased:
  - ✓ Izod Impact *(-Negative)*
  - ✓ Mold shrinkage *(+Positive)*
  - ✓ Thermal expansion *(+Positive)*
  - ✓ Part cost *(+Positive)*

MATERIALS USED IN THIS PROGRAM:

- Montell Homopolymer Polypropylene ProFax 6323 (12 MFI)
- Amoco Polypropylene 1016 (5 MFI)
- Calcium Carbonate
  - Omyacarb F (Untreated)
  - Omyacarb FT (Treated)
- Struktol ZB30A
  - Fatty acid derivative
- Struktol ZB30B
  - Fatty acid derivative
- Struktol ZB31/1
  - Fatty acid derivative

FORMULATIONS:

Material	Treated	Untreated	A	B	C	D	E	F
PP Resin	100	60	59.6	59.4	59.2	59.6	59.4	59.2
$CaCO_3$ (F or FT)		40	40	40	40	40	40	40
ZB30A			0.4	0.6	0.8			
ZB30B						0.4	0.6	0.8
ZB31/1								
TOTAL	100	100	100	100	100	100	100	100

Material	G	H	I
PP Resin	59.6	59.4	59.2
$CaCO_3$ (F or FT)	40	40	40
ZB30A			
ZB30B			
ZB31/1	0.4	0.6	0.8
TOTAL	100	100	100

NOTE: All formulations with additives use untreated $CaCO_3$

LABORATORY EXPERIMENT:

Data generated is based on compounding on a Brabender PL2000 torque rheometer using the bowl mixer equipped with cam blades at 180°C and 70 rpm. The total time of the mixing cycle was 800 seconds.

Capillary rheometer testing was performed on a Shimadzu CFT-500C equipped with a 10 mm by 1 mm diameter die and set at 200°C. A variety of shear rates were used to give a complete viscosity picture.

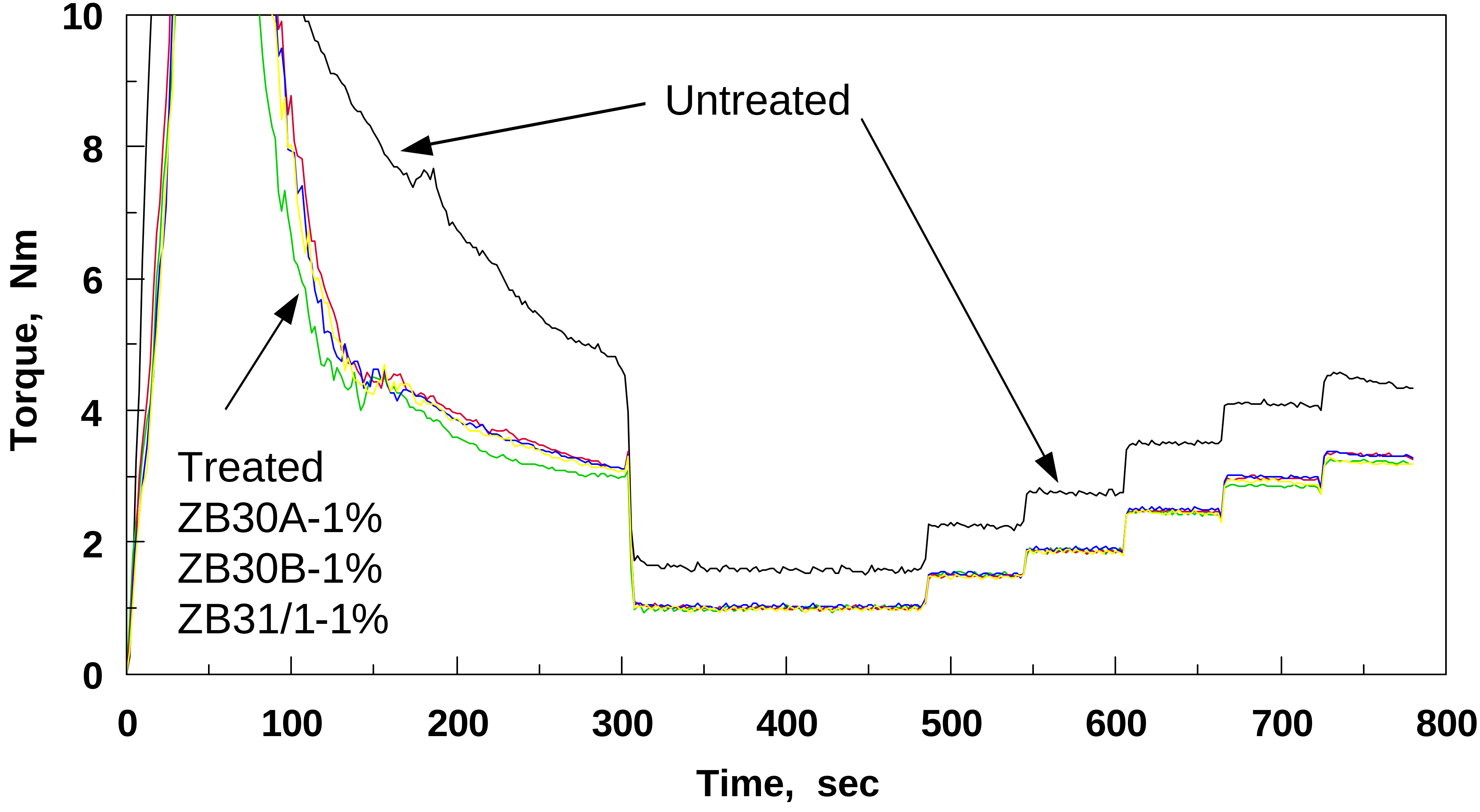
Physical property data was generated using injection molded test specimens prepared on a Cincinnati Milacron 33 ton lab molding machine:

Color measurements were taken on injection molded specimens using a Minolta Spectrophotometer.

Thermal stability measurements were made on a Mettler Toledo DSC at 10°C/min.

MIXING CURVES FOR 40% $CaCO_3$ FILLED COMPOUNDS

ProFax 6323



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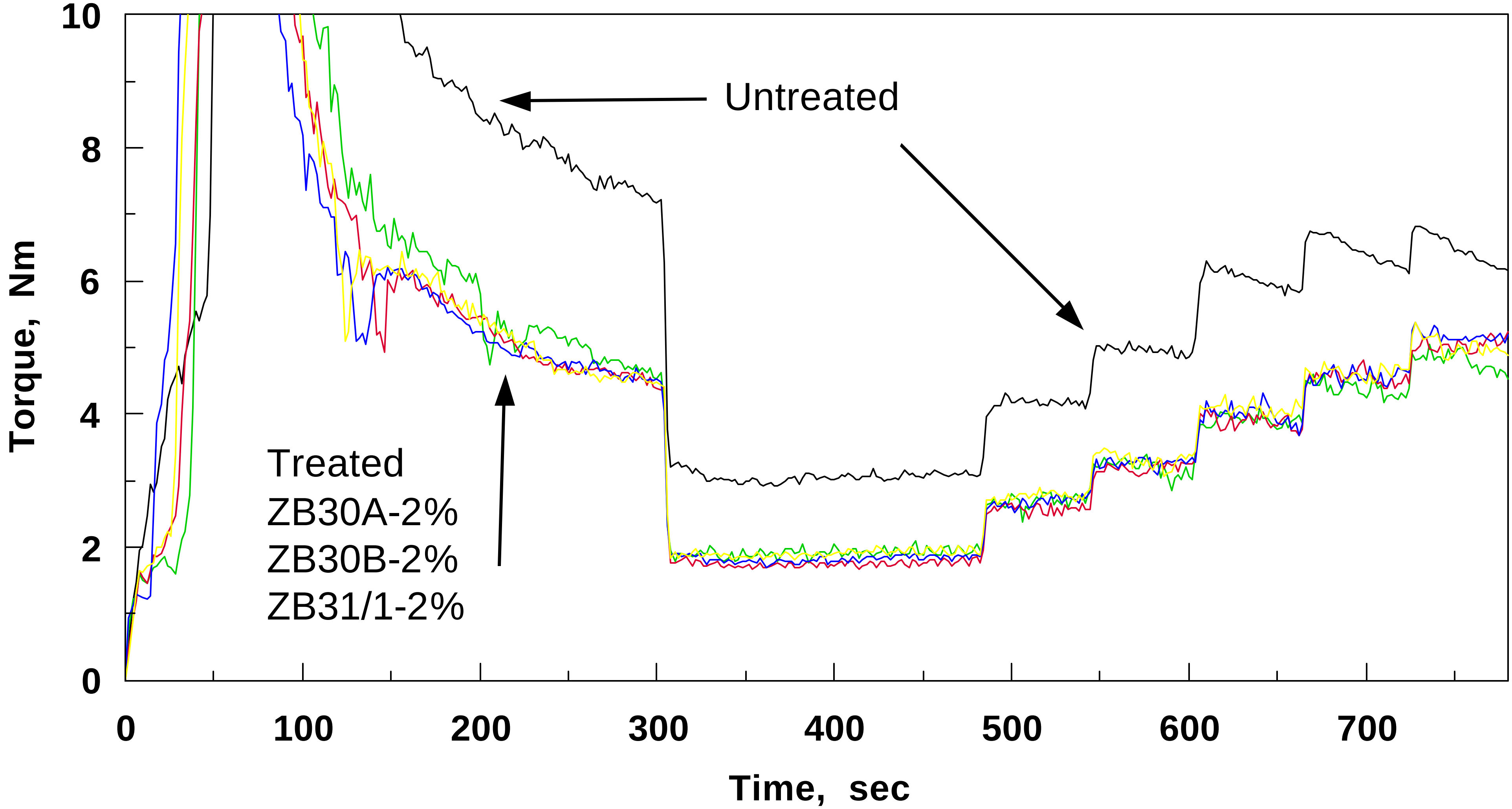
MIXING CURVES FOR 40% $CaCO_3$ FILLED COMPOUNDS

Amoco 1016

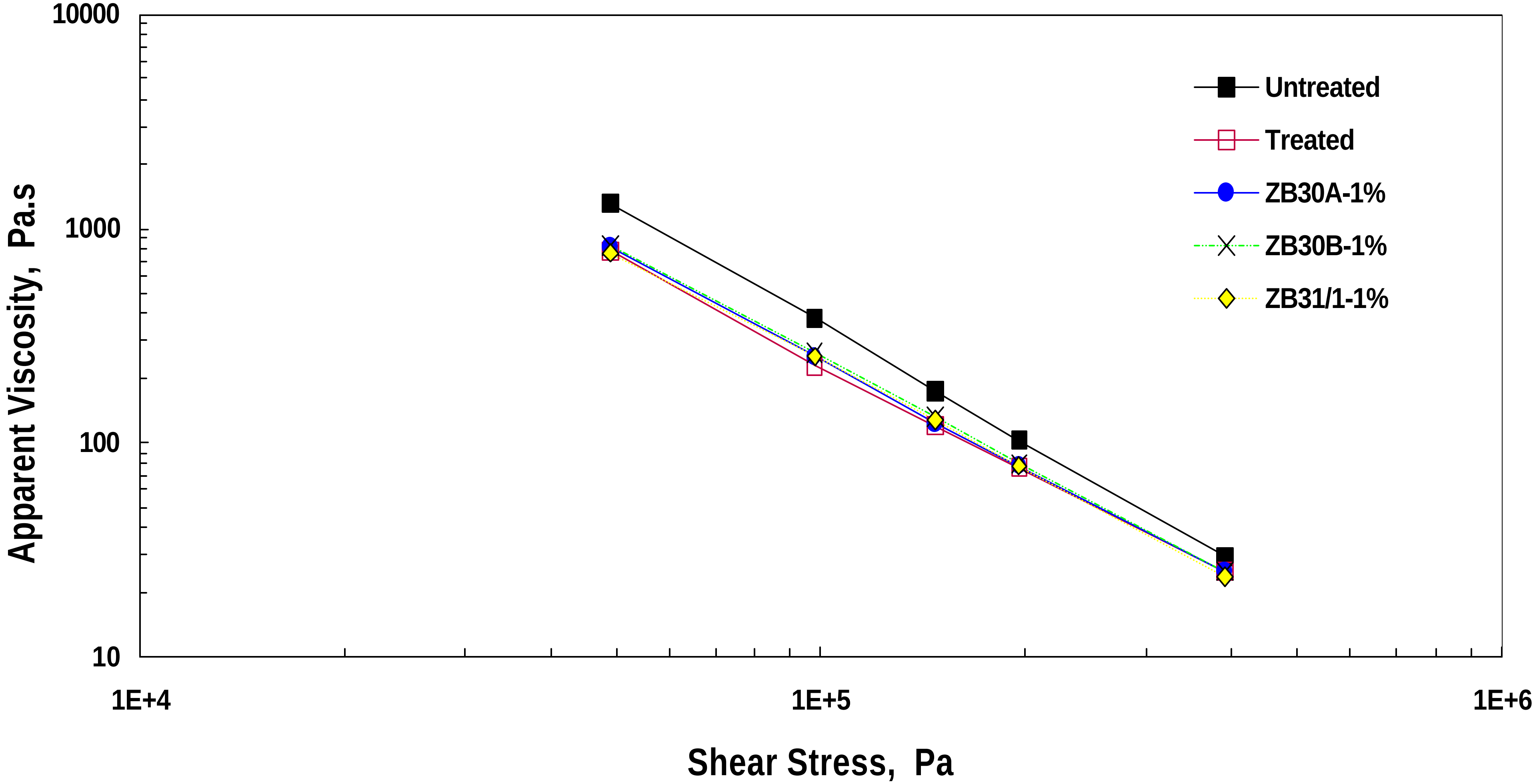


MIXING PROPERTY RESULTS:

- Untreated $CaCO_3$ with no additives showed higher torques throughout the mixing cycle and the resulting compound displayed poor dispersion with residue $CaCO_3$ left in the bowl.
- The treated $CaCO_3$ showed improved wetting characteristics resulting in lower torques throughout the mixing cycle. The resulting compound displayed good dispersion and color.
- The untreated $CaCO_3$ with fatty acid derivatives added showed wetting and mixing characteristics typical of treated $CaCO_3$. The resulting compound displayed good dispersion and color.

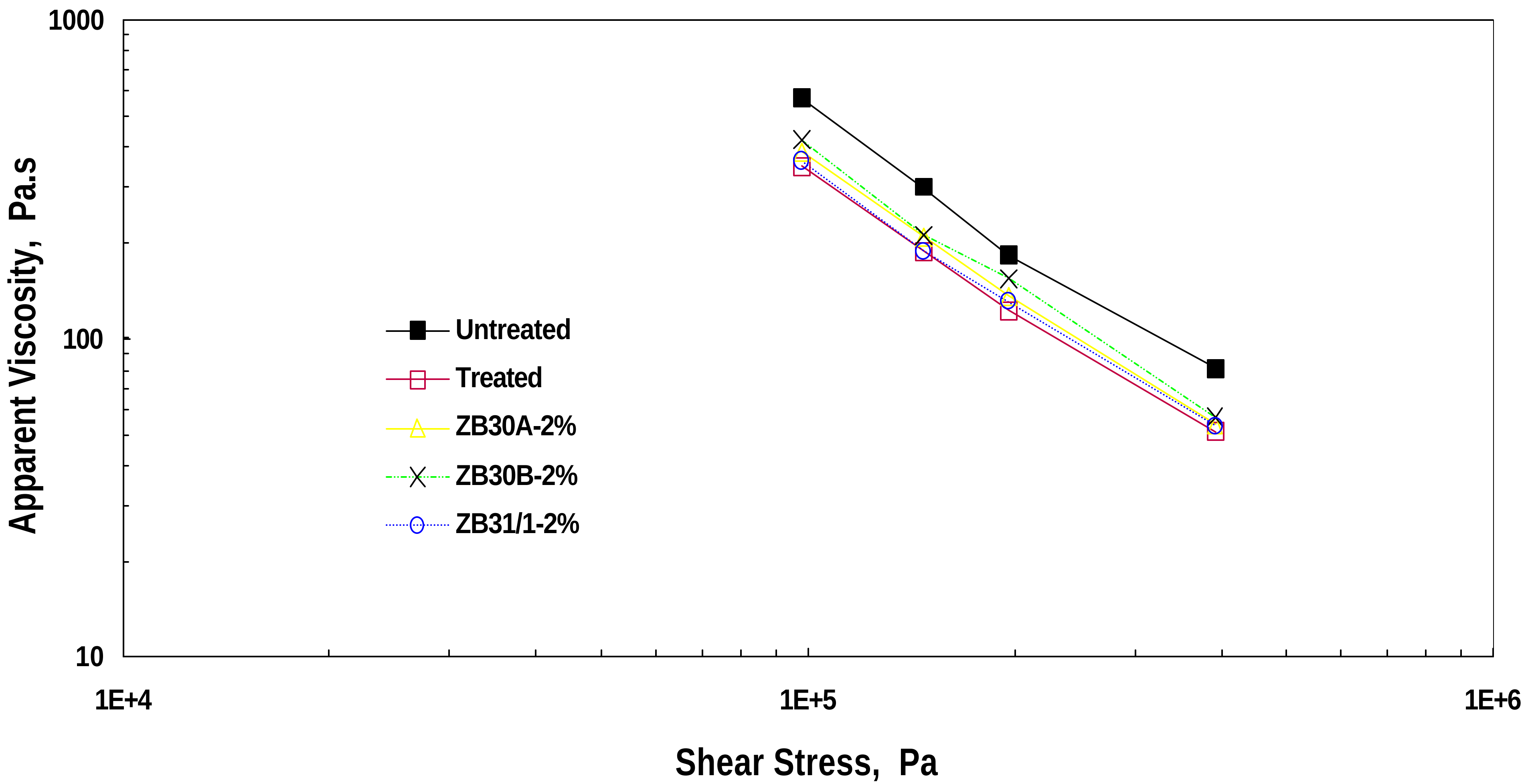
VISCOSITY AND SHEAR STRESS AT 200°C

ProFax 6323 Compounds



VISCOSITY AND SHEAR STRESS AT 200°C

Amoco 1016 Compounds



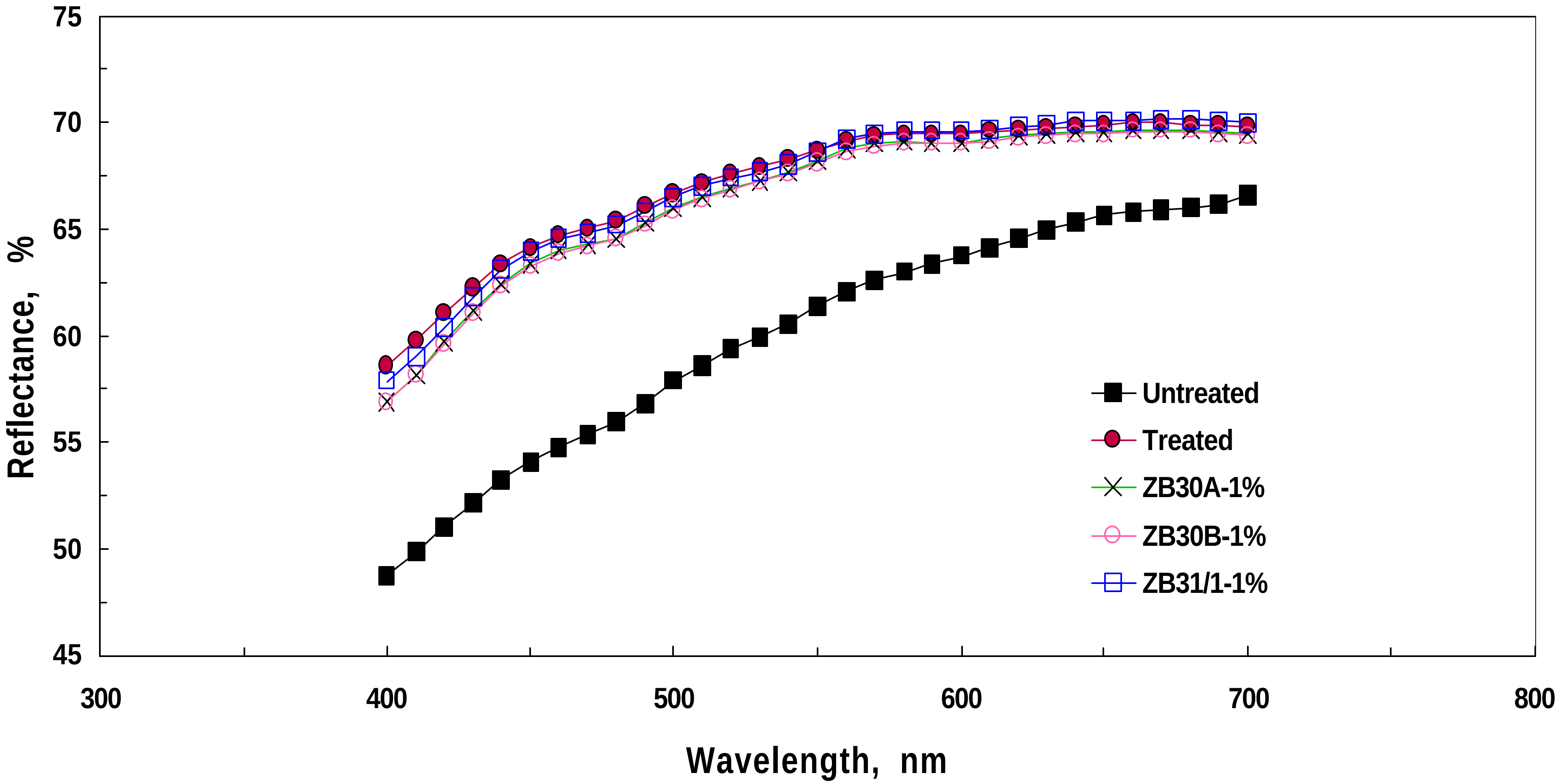
VISCOSITY TESTING RESULTS:

- Untreated $CaCO_3$ with no additives had higher melt viscosities across the shear range when compared to treated $CaCO_3$.
- The untreated $CaCO_3$ with fatty acid derivatives added had melt viscosities comparable to treated $CaCO_3$ across the shear range.

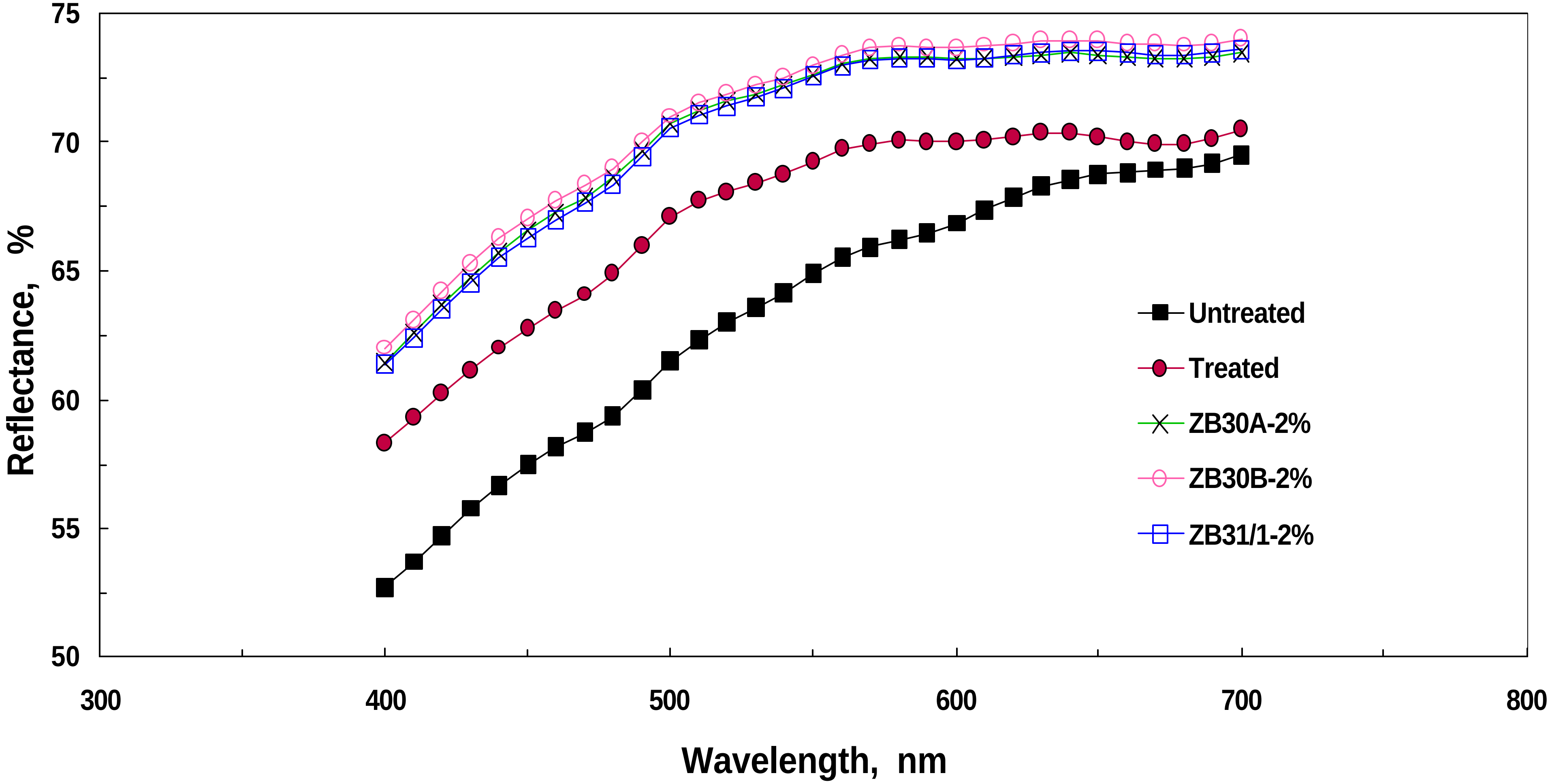
SPECTROPHOTOMETER DATA, D65/10°

Description	L*	a*	b*	C*	h
ProFax 6323					
Untreated	85.2	0.6	4.5	4.6	82.4
Treated	87.2	-0.3	3.5	3.5	94.3
ZB30A - 1%	88.1	-0.3	3.5	3.5	95.0
ZB30B - 1%	87.8	-0.3	3.6	3.6	94.0
ZB31/1 - 1%	88.3	-0.3	3.3	3.3	95.0
ZB30A - 1.5%	88.2	-0.3	3.3	3.3	95.6
ZB30B - 1.5%	88.1	-0.3	3.4	3.4	95.0
ZB31/1 - 1.5%	88.2	-0.3	3.4	3.4	95.1
Amoco 1016					
Untreated	84.2	0.7	6.5	6.5	84.2
Treated	86.4	-0.5	5.2	5.2	96.0
ZB30A - 1.5%	87.6	-0.6	5.5	5.5	96.5
ZB30B - 1.5%	88.0	-0.4	5.0	5.0	95.1
ZB31/1 - 1.5%	87.7	-0.5	4.7	4.7	96.1

REFLECTANCE BY SPECTROPHOTOMETER FOR ProFax 6323 COMPOUNDS, D65/10 degree



REFLECTANCE BY SPECTROPHOTOMETER FOR AMOCO 1016 COMPOUNDS
D65/10 degree



COLOR TESTING RESULTS:

- Untreated $CaCO_3$ with no additives gives a more yellow/grey compound when compared to treated $CaCO_3$.
- The untreated $CaCO_3$ with fatty acid derivatives gives a much whiter compound comparable with and in some cases better than the treated $CaCO_3$.
- In the 5 MFI PP compound, reflectance data shows a much whiter compound in the untreated $CaCO_3$ with fatty acid derivatives.

FLEXURAL PROPERTIES AND IZOD IMPACT STRENGTH:

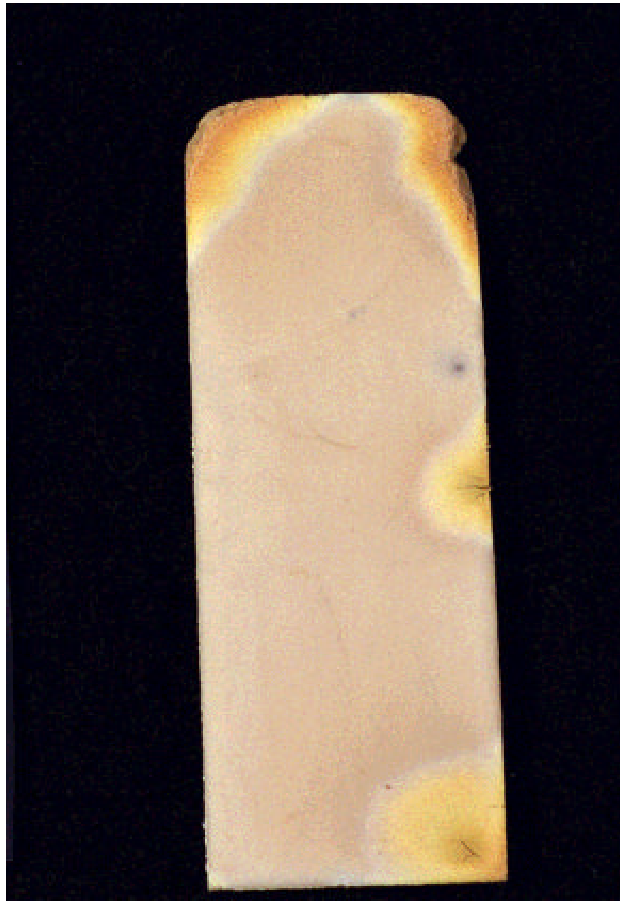
	Flex Strength, MPa	Flex Modulus, MPa	Notched Impact, J/m	Unnotched Impact, J/m
ProFax 6323				
Untreated $CaCO_3$	44.1	2191	75	641
Treated $CaCO_3$	48.6	2795	150	No Break
ZB30A - 1%	45.7	2274	171	No Break
ZB30B - 1%	49.0	2640	171	No Break
ZB31/1 - 1%	45.9	2265	176	No Break

MELT TEMPERATURE AND DEGRADATION BY DSC:

	T_m, °C	T_{deg}, °C
Unfilled ProFax 6323	166	246
Untreated $CaCO_3$	168	237
Treated $CaCO_3$	166	231
ZB30A - 1%	167	248
ZB30B - 1%	167	248
ZB31/1 - 1%	168	247

OVEN AGING DEGRADATION (158 hrs. AT 150°C:

ProFax 6323
+ Untreated $CaCO_3$ (40%)



ProFax 6323
+ Treated $CaCO_3$ (40%)

ProFax 6323
+ Untreated $CaCO_3$ (40%)
+ Fatty Acid Derivative (0.4%)

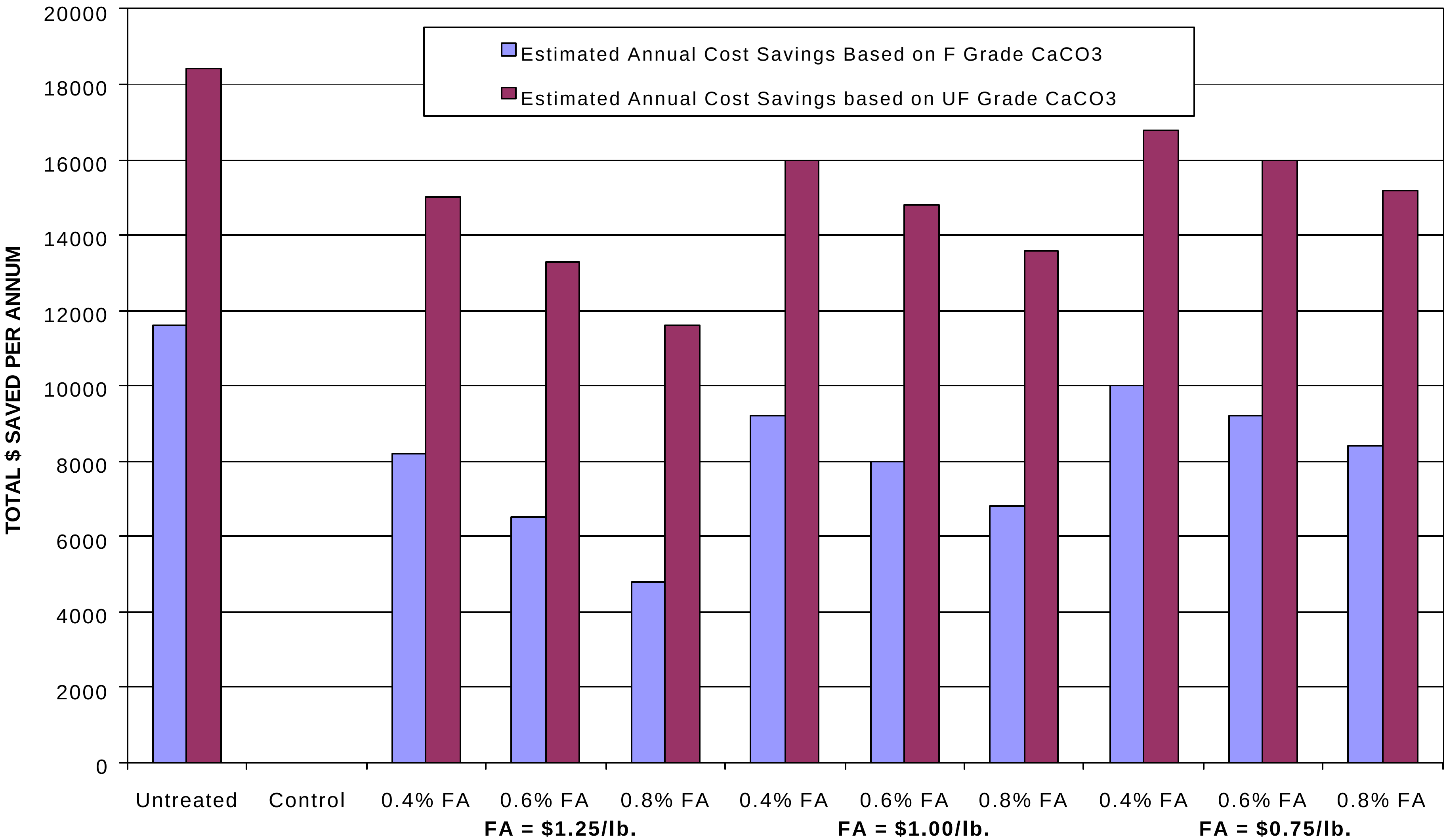
ECONOMICS:

- Price estimations used:
  - Untreated, F Grade $CaCO_3$ = $0.093/lb.
  - Treated, F Grade $CaCO_3$ = $0.122/lb.
  - Untreated, UF Grade $CaCO_3$ = $0.134/lb.
  - Treated, UF Grade $CaCO_3$ = $0.180/lb.
  - Polypropylene, generic = $0.400/lb.
  - Fatty Acid Additive analyzed at $1.25, 1.00 and 0.80/lb.

- Compound costs based on the formulations used in this study
- No change in output rates or other production costs

COST SAVINGS REALIZED USING FATTY ACID DERIVATIVES VS. TREATED CONTROL

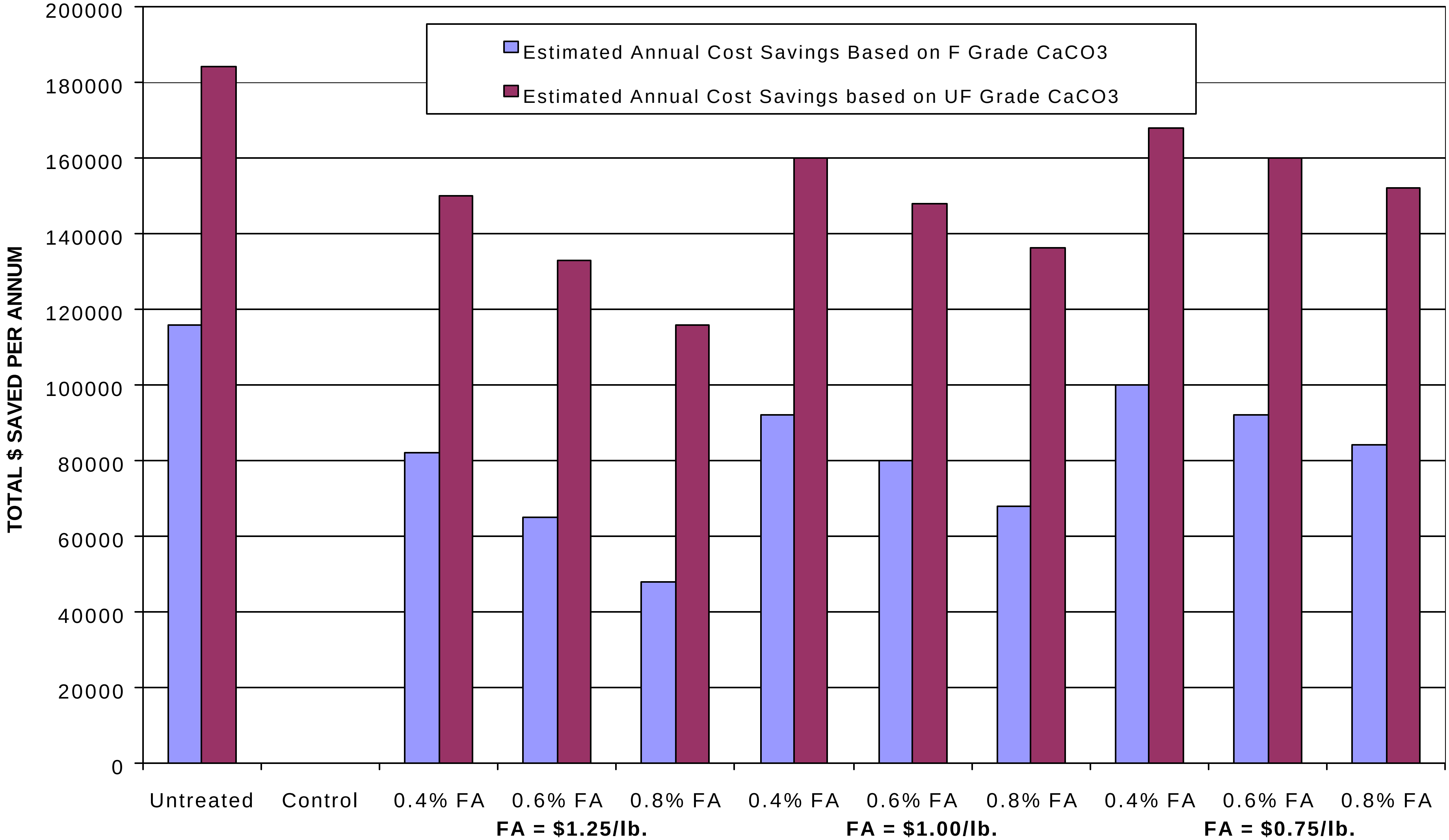
1 MM LB. PRODUCTION RUN



Quality Additives for Performance

COST SAVINGS REALIZED USING FATTY ACID DERIVATIVES VS. TREATED CONTROL

10 MM LB. PRODUCTION RUN



Quality Additives for Performance