

August 10, 2005

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RE: Processability of Polyphenylene Sulfide Nanocomposites

Dear Dr. Ibeh:

I am glad to inform you that the final paper for the above project has been completed and is ready for your approval.

This paper will inform the reader of the Processability of Polyphenylene Sulfide Nanocomposite, using Ryton R4 200NA as the matrix with the addition of Nanocor Nanoclay I.30E at 1, 3, 5, 7, and 10% levels; Moreover, the future experimentations with silicon carbide and carbon nanofibers and other nanotechnologies of nano-filled high temperature thermoplastic.

Sincerely,
Derrick Lamm

pc: Derrick Lamm, undergraduate student, Plastics Engineering Technology,
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PSU/ONR – REU/RET
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Processability of Polyphenylene Sulfide Nanocomposites

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Summary:

This paper shows the use of the Haake Torque Rheometer Rheocord System 40 to compare the processability measured in mg (metergrams) of polyphenylene sulfide nanocomposites. Chevron Phillips Ryton R4 200NA was the matrix and Nanocor Nanomer I.30E nanoclay was the filler and 0,1,3,5,7,and10% levels were tested and compared using graphs that showed the torque(mg) vs. time(sec). A reading every 6secs was taken during the test and was the data used for comparison. The results show that 1% and 3% are the best suited for lowering the processability of polyphenylene sulfide nanocomposite.

Introduction:

Over the past eighteen months, plastic nanocomposite applications have gained commercial attention, due to the efforts of resin manufacturers, compounders and masterbatch producers who now offer user-friendly products. Although applications vary widely, they principally exploit the technology's contributions in the areas of gas barrier, reinforcement, and flame retardancy. Now especially, with the importance of non-halogen flame retardant compounding due to new world wide environmental issues nanocomposites have a market niche to fill. Nanocomposites differ from traditional plastic composites in that they provide these properties with little impact on part weight and they do so without great processing problems.

Nanomers are surface modified montmorillonite minerals available for a large range of polymer resins from commodity polyolefins to specialty polyamides. Incorporation into these resins forms a nanocomposite plastic. Because Nanomers are used at low addition levels, significant property improvement is achieved with lighter part weight. Nanocomposite plastics are potent char formers, making them a valuable tool in creating improved fire retardant materials. In fluid resin systems Nanomer rheology is similar to that of fumed

silica, and in addition to traditional property improvement, it carries the benefit of providing flow control when used in thermoset resin formulations. Commercial nanocomposite plastics are emerging rapidly. These include nylon 6 and polypropylene for packaging and injection molded articles, semi-crystalline nylon for ultra-high barrier containers and fuel systems, epoxy electrocoat primers and high voltage insulation, unsaturated polyester for watercraft lay-ups and outdoor advertising panels, and polyolefin fire retardant cable, electrical enclosures and housings.

Definitions:

compatibilization: the process of surface modifying a nanoclay so that it is attracted to and will disperse in resin matrices. The two most common compatibilization classes are onium ion modification and ion-dipole interaction

exfoliation: a process wherein packets of nanoclay platelets separate from one another in a plastic matrix. During exfoliation platelets at the outermost region of each packet cleave off, exposing more platelets for separation. Nanoclay compatibilization is essential for exfoliation

intercalant: (surface treatment) containing an ammonium or phosphonium functional group. The groups modify a nanoclay surface by ionically bonding to it, converting the surface from a hydrophilic to an organophilic species.

intercalate: a clay-chemical complex wherein the clay gallery spacing has increased, due to the process of surface modification. Under the proper conditions of temperature and shear, an intercalate is capable of exfoliating in a resin matrix.

montmorillonite clay: the most common member of the smectite clay family. Montmorillonite is generally referred to as “nanoclay”. It is also the most common material used in plastic nanocomposites

nanoclay: a clay from the smectite family. Smectites have a unique morphology, featuring one dimension in the nanometer range.

nanocomposites: new class of plastics derived from a highly refined form of nanoclay that disperses in plastic resins. These nano-sized particles are composed of montmorillonite minerals

nanometer: a unit of measure. One nanometer corresponds to a length which is one-billionth of a meter, or about one ten-thousandth the diameter of a human hair.

Nanomer® nanoclays: surface modified montmorillonite clays, or masterbatches containing modified clays, that are utilized to make a nanocomposite. Nanomer nanoclays may be generically referred to as "intercalates" in patent literature

onium ion modification: the formation of a clay-chemical complex using an intercalant (surface treatment) containing an ammonium or phosphonium functional group. The groups modify a nanoclay surface by ionically bonding to it, converting the surface from a hydrophilic to an organophilic species.

organophilic: a chemical or molecular orientation favoring the attraction of hydrocarbons or materials which are miscible in hydrocarbons. Organophilic materials are characterized by weak dipole moments. They are basically immiscible in water.

platelet: general shape of a montmorillonite particle, consisting of a sheet-like structure where the dimensions in two directions far exceed the particle's thickness.

thermoplastic: plastic that softens when exposed to heat and returns to its original condition when cooled to room temperature.

Problem Statement

Exfoliate Chevron Phillips Ryton R4 200NA with Nanocor Nanoclay I.30E and compare torque used for processing by changing the levels of I.30E introduced to nanocomposites.

Historical Background

1907 Bakelite created by Leo Baekeland

1915 Pyrex (borosilicate glass)

1930 Synthetic rubber developed by Wallace Carothers at DuPont

1930s Glass fibers become commercially viable

1933 Polyethylene discovered
1934 Nylon
1936 Polymethyl acrylate discovered and leads to Plexiglass (polyvinyl methacrylate)
1938 DuPont discovers Teflon
1946 Tupperware discovered by Earl Tupper a chemist at DuPont
1950s Silicones
1953 Dacron or polyethylene terephthalate revolutionizes textiles industry
1953 Karl Zeigler creates high-density polyethylene by new method
1964 Carbon fiber developed
1977 Electrically conducting organic polymers discovered
1986 Synthetic skin
1990s Nanotechnology

Objectives

Exfoliate Nanocor Nanoclay I.30E into Chevron Phillips Ryton, Polyphenylene Sulfide, using Haake Torque Rheometer and comparing the torque values required for exfoliation.

Future objectives:

Exfoliate Nanocor nanoclay I.30E, Nanocor nanoclay I.34TCN, silicon carbide, and carbon nanofibers within Ryton R4 200NA using the twin screw extruder and pelletizing. Exfoliated nanocomposite pellets will be injection molded, making ASTM test bars. Mechanical test will be performed such as tensile using the Instron Universal Testing machine and impact using the Izod Impact Testing Machine. The Flammability will be tested using a cone calorimeter for precise and accurate measurements of flame retardancy. A sonicator will be used along with a Chevron Phillips Ryton powder PR26 to test dispersion and emulsion techniques of and analyzed using the Atomic Force Microscope.

Impact Statement and Significance of Study

This study will show that nanoclay fillers within polymer matrix will lower the processability or ease of processing compared to typical fillers that will raise the processability. On a budgeted operation in industry every penny makes a drastic impact on the overall cost of producing a part and lowering processability will make the energy levels used to process lower which in turn means more profit.

Scope

The overall scope of the project includes several nanotechnologies and several nanomaterials.

The advent of nanotechnology holds the promise of developing new materials with processability, properties, and performance far exceeding that of conventional materials. Use of these revolutionary materials in military, aerospace, and power propulsion systems could result in significant improvements in component durability, safety and performance, as well as reduced weight. Nanoclays can be used with commercial and some engineering thermoplastic along with thermosetting polymers and allows conventional fillers used by industry to be replaced and with technology becoming smaller micro molding is a industry that nanoclay technology would be used. New advances with silicon carbide offer the possibility of developing nanotube reinforced metals and ceramics with higher temperature capabilities. Polymer/clay nanocomposites have been developed with significantly better high temperature stability and mechanical properties than conventional composite materials. Carbon nanofibers are used as reinforces that aid mechanical properties far beyond virgin polymer matrix parts. Carbon nanofibers are becoming longer and longer as technology and knowledge of carbon nanofibers expands each year and carbon nanotubes are a growing interest for reinforcement. Exfoliation is primary dispersion technique used with nanotechnology but emulsion polymerization offers a viable, flexible route for nanocomposite fabrication from nanoscale spheres, rods and plates. Combining emulsion generated polymer particles that are ionically stabilized in aqueous solution with a better dispersion

of nanoparticles using sonicator technique results in an interfacial exchange reaction and co-precipitation.

Literature Review

Polymer Nanocomposites Open a New Dimension for Plastics and Composites. AMPTIAC Quarterly. Vol 6 #1.

The nanoscale is the new frontier in materials and nanoscience and technology allows for new material combinations to advance technology. Polymers are the main direction of nanotechnologies and nanocomposites will usher a new era in materials development. Polymer Nanocomposites, PNC, offer conventional alternatives for fillers used in conventional filled polymers and polymer blends. PNC technology is not based solely on mechanical enhancement of resin but rather adding properties not present in the resin without sacrificing its inherent processibility and mechanical properties. Currently, exfoliation of layered silicates and high performance resins are the most heavily investigated PNCs by industrial, government and academic institutions world wide. In-situ routes to PNCs are based on the creation of the nanoelement within the polymer matrix by chemical means or phase separation with the polymer matrix providing the template within which the nanoelement is formed. Layered silicates are the most commonly used inorganic nanoelements in PNC research to-date. Layered silicates possess the same structural characteristics as minerals talc and mica and are commonly comprised of hydrated aluminosilicate. In general, layered silicate PNC produce glassy modulus enhancements of one to two times and rubbery modulus increases of 5-20 fold. Additionally, reduced thermal expansivity (CTE), matrix swellability, gaseous permeability and flammability upon 1-5% addition of exfoliated layered silicates. One of the important resins researched with layered silicates is phenolics. The objective of PNC fabrication by exfoliation methodology is to uniformly disperse and distribute the inorganic filler, initially comprised of aggregates of the nanoparticles, within the polymer. The final PNC structure results from the transformation of the initially microscopic heterogeneous system to a nanoscopically homogenous system. There are

four approaches to develop PNC via exfoliation: solution processing, mesophase mediated processing, in-situ polymerization and melt processing. Upon incorporation of the nanoelements in thermoset resins the surface functionization must be carefully chosen to control polymerization rates and initiation points such that separation of the aggregate of nanoelements occurs before or during polymerization. This is because of the extent of cross-linking reactions determines the gel-point of the matrix, and ultimately the extent of exfoliation, hence the final PNC morphology.

By: Vaia, Dr. Richard A.

Improving the Fire Performance Characteristics of Composite Materials for Naval Applications. AMPTIAC Quarterly. Vol.7 #3.

The principle advantages of composites include higher stiffness to weight ratio, endurance under cyclic loading, and resistance to chemical attack. There is a growing interest in composite materials for Naval applications such as deckhouses, lightweight foundations, and masts. The Navy recognizes that composites are a good substitute for the current material used for surface ship application. Brominated vinyl-ester resin with glass reinforcement is the current material used. The development of new materials are phthalonitrile by NRL, modified phenolics by General Dynamics' Electric Boat Corp and epoxy, cyanate-ester based on bisphenol C by Federal Aviation Administration, Polyhedral oligomeric silsesquioxane (POSS) based resins, nanoclay reinforced vinyl-esters and more. Phthalonitrile Resin possesses excellent attributes for Navy ships like long term mechanical properties up to 700degF and a Tg of 932degF. This material uncured has a low melt viscosity that allows it to be processed using a transfer molding process. POSS is manufactured by Hybrid Plastics Inc. and is called a hybrid. POSS is compatible with existing resins and improves the thermal, dielectric and mechanical properties of traditional polymers and is also compatible with existing manufacturing processes. POSS nanomaterials are biocompatible, recyclable, non-flammable, and competitively priced. The best

way to test material is the measurements of heat release rates in small scale cone calorimeters.

By: Sorathia, Usman

Mechanical and Flammability Resistance Properties of Extrusion Enhanced ABS/PC Nanocomposites

ABS and polycarbonate are renowned for their low temperature properties whereas their blends have enhanced flammability resistance and lower cost properties. The use of nanoparticles in combination with polymeric materials to create plastic nanocomposites (PNCs) is a current trend. The infusion of these nanoparticles in polymeric matrix is typically carried out via sonication. In this project, we explore the alternative but very viable process of extrusion as a nanoparticle infusion process, and present the results obtained for the mechanical and flammability resistance properties of nanoclay-infused ABS/PC blends. Torque Rheometry data indicate that processability is enhanced with small amounts of nanoclay up to the 4.0 PHR level. Optimal mechanical properties are attained at the 3.0 PHR nanoclay level whereas flammability resistance properties increase with increase in nanoclay level.

Christopher C. Ibeh, Nathan Baker, Derrick Lamm, Shyang Wang,
Derek Weber, Jon Oplotnik

Processability of Polyphenylene Sulfide Nanocomposites

The experiment was to compare Chevron Phillips Ryton R4 200NA the Nanocor nanoclay I.30E that is an onium ion surface modified montmorillonite mineral. It is designed to be easily dispersed into amine-cured epoxy resins to form nanocomposites, but was suggested by Nanocor representatives that it was the best suited for high temperature thermoplast application. With use of epoxy a number of properties were improved, including modulus, Tg and chemical

resistance. Amine-cured epoxy nanocomposites with 10 ~ 20 phr of Nanomer I.30E nanoclay exhibit significantly increased glass transition temperatures 10 to 20 °C (DMA method). The energy required to plasticize R4 200NA is high compared to general commodity thermoplastic with a Tg relatively high and a CUT higher the processing temperature for the material is 600°F or 315°C and typical filler will increase the energy needed to plasticize the polymer.

Methodology

Equipment

AEC Whitlock Dryer
Haake Torque Rheometer System 40 Rheocord

Materials

Chevron Phillips Ryton: R4 200NA
Nanocor Nanoclay: I.30E

Procedure

AEC Whitlock Dryer

Switch on circuit breaker and main power
Ensure air hose is connected to machine
Ensure all air hose lines are open
Place desired amount of material in pillowcase
Open door latches and place bagged material in hopper
Turn on power to main system
Set temperature for hopper: Ryton R4 200NA 250°F for 2hrs

Haake Torque Rheometer System 40 Rheocord

- Turn on Drive Unit
- Turn on Processing Unit
- Calibrate Haake Torque Rheometer
- Enter "Setup"
- Enter "Title"

- Enter “Temperature” from zone 1(315°C) cooling (N); zone 2(315°C) cooling (Y); zone 3(315°C) cooling (N); zone 4(0°C) cooling (N). All zone are non-programmable.
- When the temperatures of the heating zones are within tolerance turn on the rotation and enter “Digital Display” make sure the Torque equals 0mg (metergrams).
- Measure 30g of Ryton R4 200NA
- Reset the time by pushing “Enter” and reenter “Digital Display”
- Place the 30g of Ryton R4 200NA in the mixing bowl and close
- Measure 30g of Ryton R4 200NA
- Open mixing bowl at 4min
- Place next sample in bowl at 5min and close
- Measure 30g of Ryton R4 200NA
- Open mixing bowl at 9min
- Place next sample in bowl at 10min and close
- Open mixing bowl at 14min
- At 15min go to “Digital Review”
- Enter start time “00”min and stop time “15”min and enter step time “06”sec
- Enter “P” to print and once complete enter “Z” to next page
- Repeat last step until finished printing data
- Turn Rotation off and remove heating plate 3 and clean
- Remove heating plate 2 and clean
- Clean screws
- Replace heating plate 2 and 3
- Enter “Digital Display”
- Turn on Rotation and make sure torque reads 0mg
- Measure 30g Ryton R4 200NA and repeat procedure until retrieved 5 sets of data
- Enter data in Microsoft Excel and make comparison

Procedure For Dispersion of Nanoclay I.30E

- Turn on Drive Unit
- Turn on Processing Unit
- Calibrate Haake Torque Rheometer
- Enter “Setup”
- Enter “Title”
- Enter “Temperature” from zone 1(315°C) cooling (N); zone 2(315°C) cooling (Y); zone 3(315°C) cooling (N); zone 4(0°C) cooling (N). All zone are non-programmable.
- When the temperatures of the heating zones are within tolerance turn on the rotation and enter “Digital Display” make sure the Torque equals 0mg (metergrams).
- Measure 30g of Ryton R4 200NA and 1% Nanoclay I.30E and mechanically mix

- Reset the time by pushing “Enter” and reenter “Digital Display”
- Place the Nanocomposite mixture in the mixing bowl and close
- Measure 30g of Ryton R4 200NA and 1% Nanoclay I.30E and mechanically mix
- Open mixing bowl at 4min.
- Place next sample in bowl at 5min and close.
- Measure 30g of Ryton R4 200NA and 1% Nanoclay I.30E and mechanically mix.
- Open mixing bowl at 9min.
- Place next sample in bowl at 10min and close.
- Open mixing bowl at 14min.
- Once at 15min go to “Digital Review”.
- Enter start time “00”min and stop time “15”min and enter step time “06”sec.
- Enter “P” to print and once complete enter “Z” to next page.
- Repeat last step until finished printing data.
- Turn Rotation off and remove heating plate 3 and clean.
- Remove heating plate 2 and clean.
- Clean screws.
- Replace heating plate 2 and 3.
- Enter “Digital Display”.
- Turn on Rotation and make sure torque reads 0mg.
- Prepare next mixture measure 30g Ryton R4 200NA and 1% Nanoclay I.30E and mechanically mix then repeat procedure until retrieved 5 sets of data.
- Enter data in Microsoft Excel and make comparison.
- Do the same procedure with Nanoclay I.30E levels at 3%, 5%, 7%, 10% or 0.9g, 1.5g, 2.1g, and 3.0g.

Results

Table 1: Processing Comparison

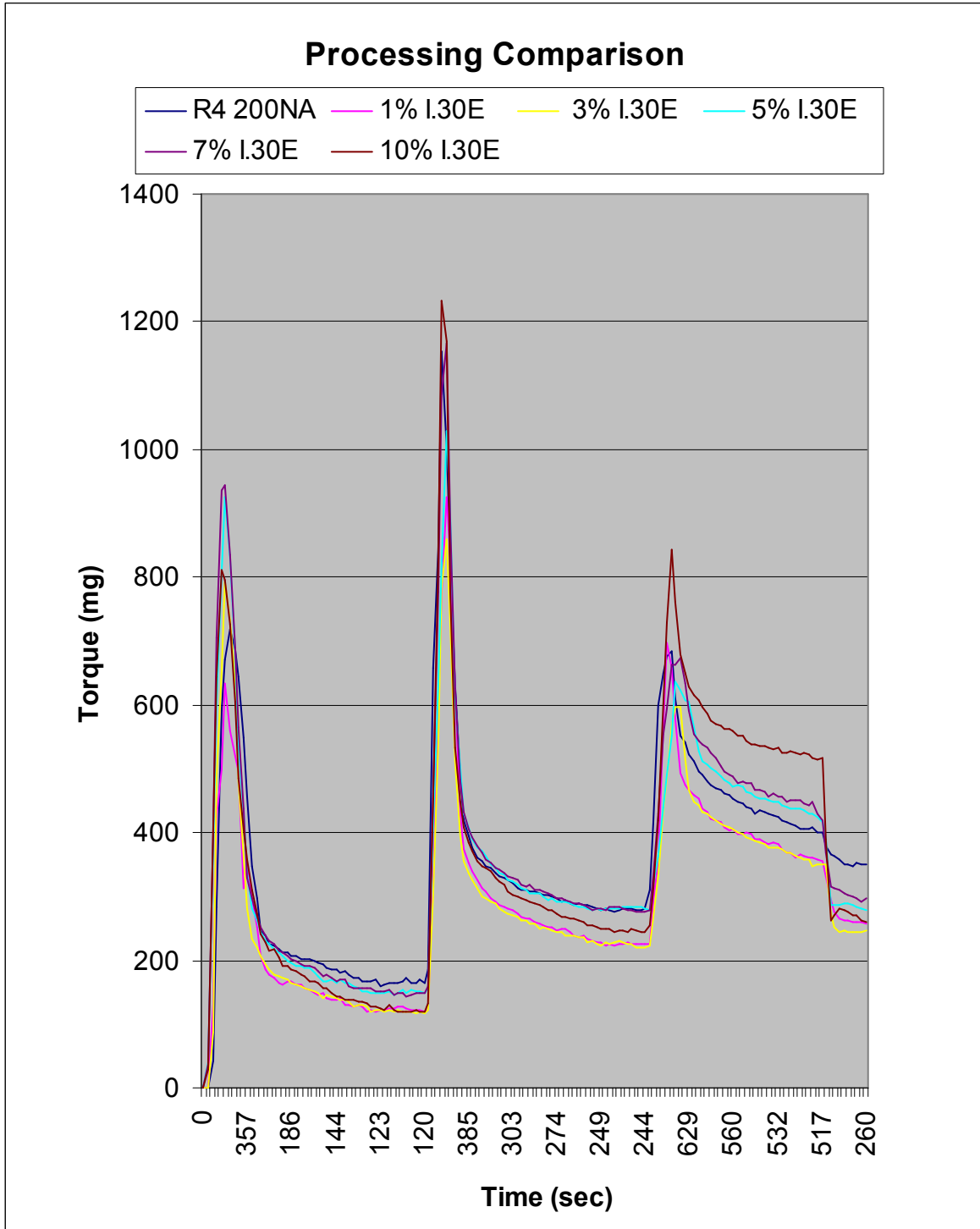


Table 2: R4 200NA 0% I.30E

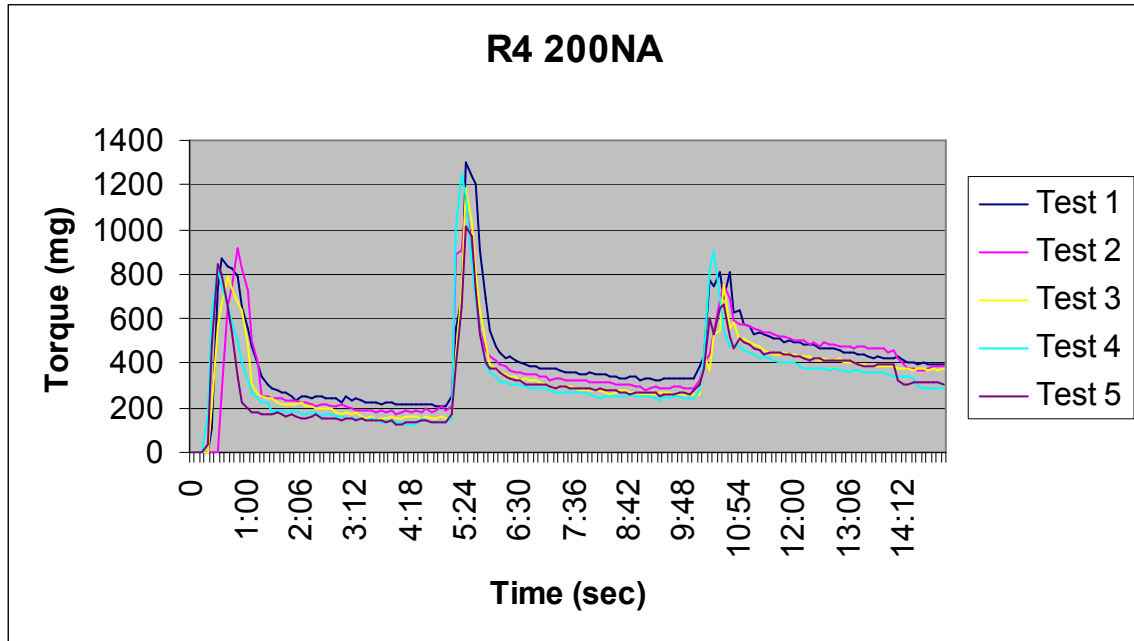


Table 3: R4 200NA 1% I.30E

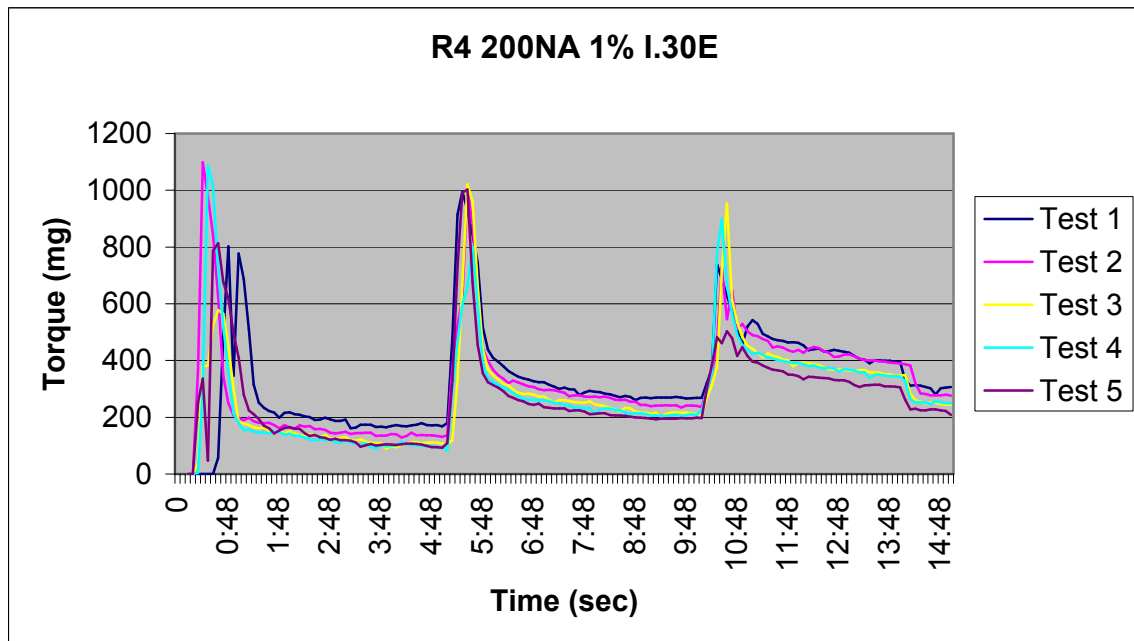


Table 4: R4 200NA 3% I.30E

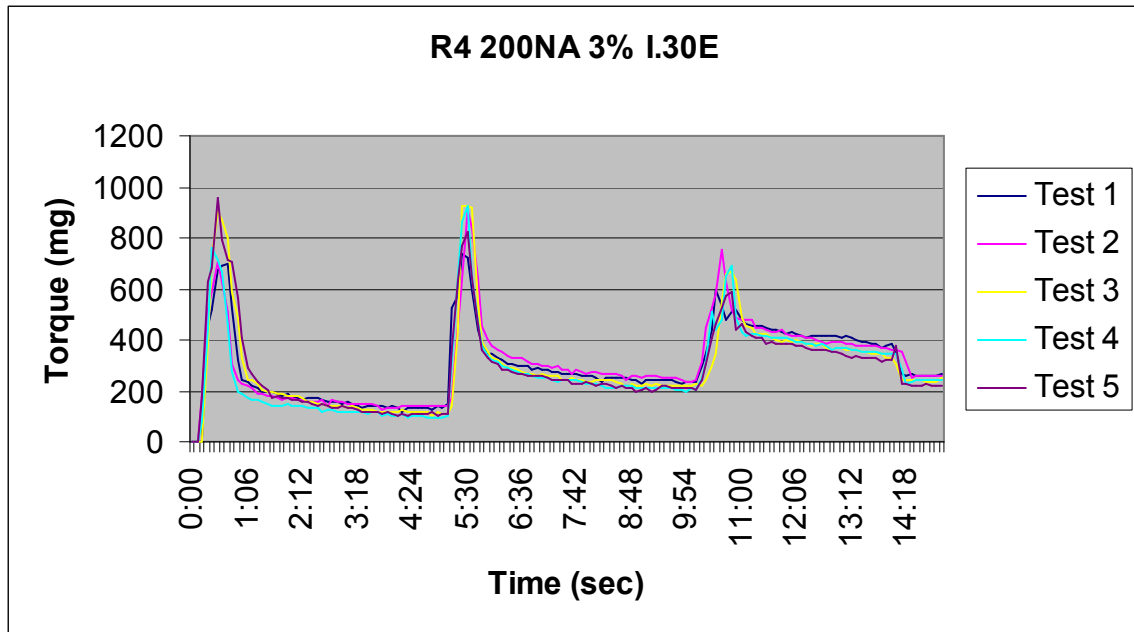


Table 5: R4 200NA 5% I.30E

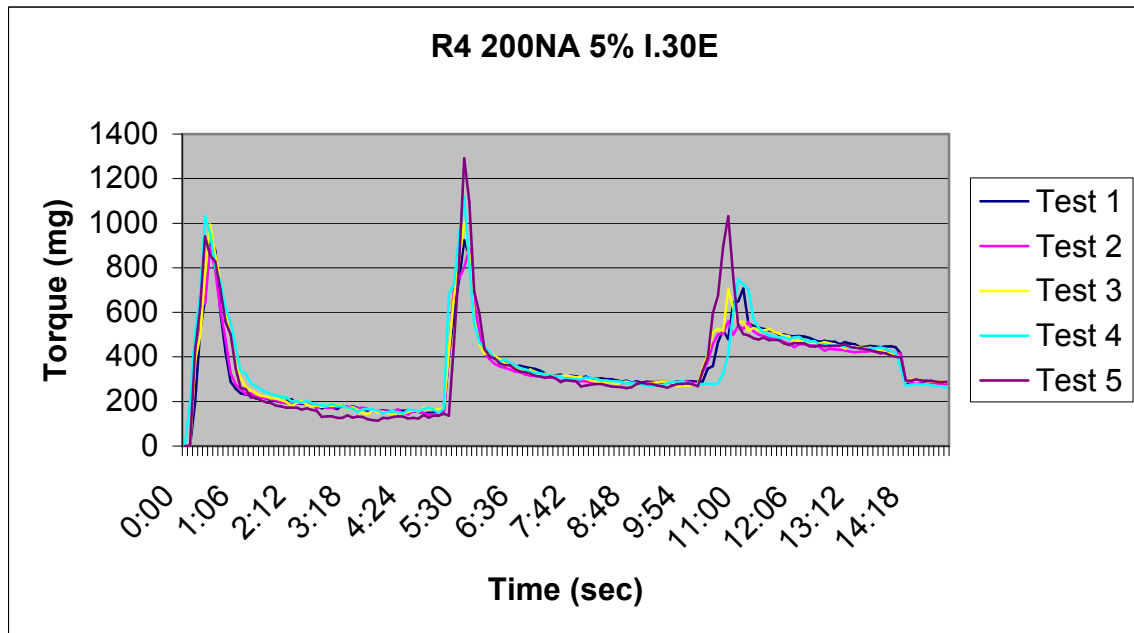


Table 6: R4 200NA 7% I.30E

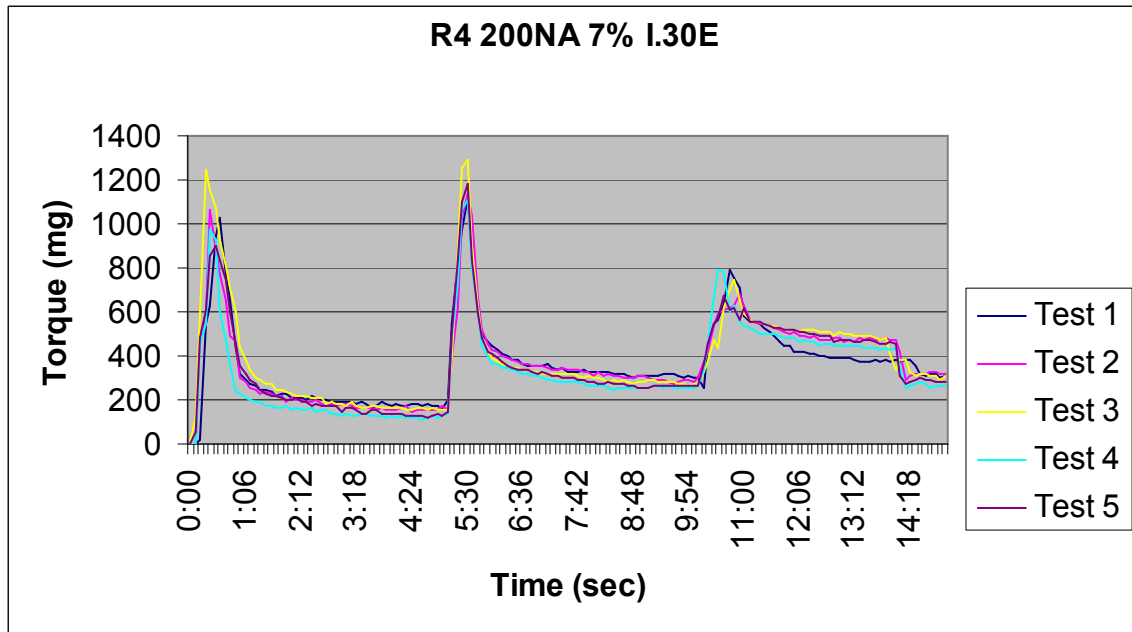
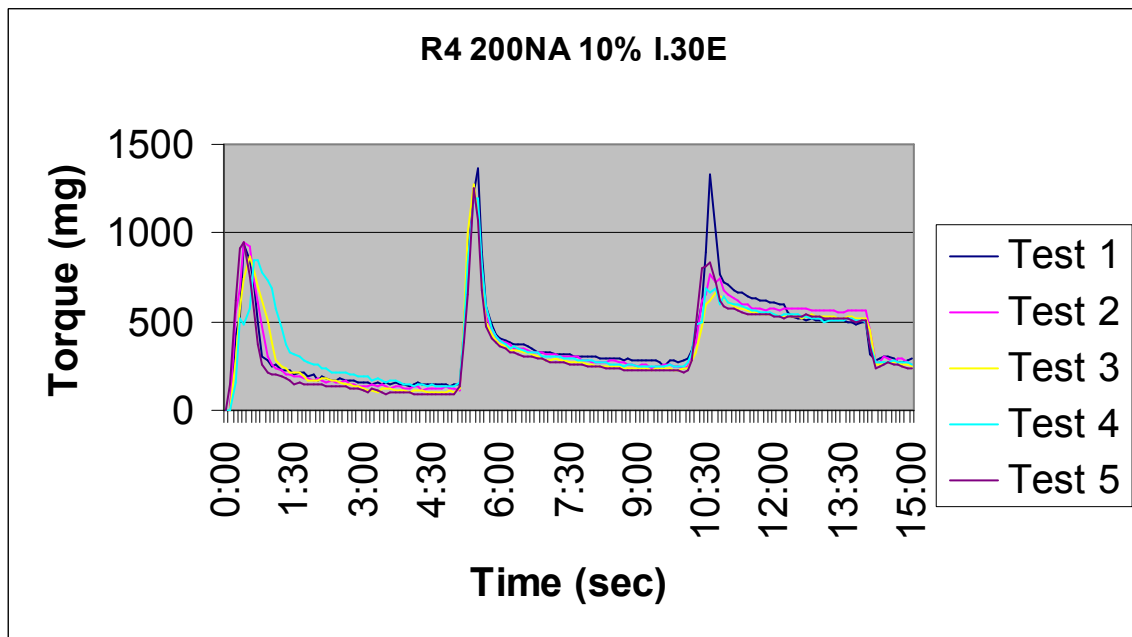


Table 7: R4 200NA 10% I.30E



Discussion of Results:

The procedure used for the experiment was a total of 15min long and gave great results compared to previous attempts. The Torque Rheometer was manufactured in 1984 and even though a primitive machine was used

Rheometer Solutions calibrated the machine and assured the data retrieved would be more than accurate. The first step was to create a procedure to would limit the amount of error to the fullest and give comparable results. The Haake Torque Rheometer Rheocord System 40 took readings every 6sec for 15min on the torque measured in metergrams. A metergram is the measure of energy or work done and the power exerted in raising one gram through the distance of one meter against gravitation. The chart starts with 0mg reading this shows that the machine has no torque and is zeroed. Then a peak appears and this is because the material is being loaded into the mixing bowl apparatus of the Torque Rheometer and force to put in bowl and force to plasticize material is large. The material relaxes as it plasticizes and slopes down. The reading that is used between 1min and 4min, 6min and 9min, and 11min and 14min shows the processability because the experiment is completely controlled in a closed environment for these three minutes. Five runs were made for each level of nanoclay dispersion and averaged and the control. The second chart of R4 200NA was used as a control or a standard in order to compare and contrast the processability of nanofiller I.30E. The averaged results were then used in the Processing Comparison chart to compare and contrast the torque levels on each polyphenylene sulfide nanocomposite. There are three stages for comparison: stage one is 1min to 4min, stage two is 6min to 9min, and stage three is 11min to 14min.

Stage 1:

The data shows that all nanofilled levels were lower than the virgin R4 200NA. The 1% and 3% being the lowest of the five test subjects then 10% and 5% and 7% are above 10%, which doesn't follow the trend. The 1% and 3% have better dispersion and allow the surface modified montmorillonite clay to ease friction between polymer chains most effectively compared to 5,7, and 10%. Why 10% doesn't follow the trend will have to be researched in future experiments over the next year.

Stage 2:

The data shows that 5% and 7% climbed and have equal or greater values as virgin R4 200NA, which makes them unsuitable for better processability of the nanocomposite. 1% and 3% are steadily lower and 10% is above these two curves. The phenomena of 10% will be further researched in future experiments.

Stage 3:

Stage 3 is the most interesting of the three stages due to the pressure created by the bulk of material in the mixing bowl and this can be show when the bowl is open at 14min and the torque values drastically decrease. 1% and 3% have very similar curves that cross through time. The virgin material is the next curve and then 5%, 7%, and finally 10% having the highest torque curve. These would suggest that the percent levels have a effect that could be calculated by use of simply equations that could be derived but this is completely theory and would need more results before any conclusion.

Conclusions:

The experiment was a success and showed that the processability can be lowered with the addition of 1% and 3% Nanomer I.30E nanoclay.

Recommendations:

To be further conclusive about the results the amount of material should be regulated at 30g per stage; for instance, stage 3 of 10% I.30E had 60g of R4 200NA and 6g of I.30E in the bowl before then application of next nanofilled mixture which totals at 90g of R4 200NA and 9g of I.30E. A more up to date Torque Rheometer would significantly change the procedure and data retrieved from experimenting with polyphenylene sulfide nanocomposites. A closed environment would also be necessary to ensure that humidity wasn't a factor.

References:

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8. Vaia, Dr. Richard A. *Polymer Nanocomposites Open a New Dimension for Plastics and Composites*. AMPTIAC Quarterly. Vol 6 #1.
9. Christopher C. Ibeh, Nathan Baker, Derrick Lamm, Shyang Wang, Derek Weber, Jon Oplotnik. Mechanical and Flammability Resistance Properties of Extrusion Enhanced ABS/PC Nanocomposites.

Appendix:

Preparation and Properties of Polyolefin Nanocomposites:

Materials:

Nanomers® I.30P, I.31PS, and I.44PA are commercial products manufactured by Nanocor, Inc. Each is an onium ion modified montmorillonite, designed for maximum compatibility and dispersion in a polyolefin matrix. Available as free-flowing powders with a mean size of 15-25 microns, they are capable of dispersing to nanoscale in twin screw compounders. In addition to the typical onium treatment, Nanomer I.31PS contains a silane-coupling agent to promote higher tensile properties. I.30P and I.31PS are film grade Nanomers and I.44PA is an engineering grade.

Screw speeds ranged from 300-500 rpm and extrusion temperatures ranged from 170~190 °C.

Nanoclay dispersion

Both XRD and TEM confirm good Nanomer® dispersion in the polyolefin matrices. XRD measures the degree of dispersion by estimating the distance between individual platelets after compounding. Table 1 presents d-spacing data for Nanomers® before and after dispersion. The increases confirm that polyolefin

host polymers have successfully interacted with Nanomer® surface treatments, promoting nanocomposite formation. TEM confirms the results

Table 1. Formation of Polyolefin Nanocomposite: Compositions and Dispersion

Polyolefin	MFI (gm/10 min)	d-spacing (Å)	
		Nanomer®	Nanocomposite
PP-PE random co-polymer	2.0	22	29
Homopolymer PP	0.45	22	28
Homopolymer PP	4.0	22	32
TPO	12	22	32

Theoretical formula and structure for montmorillonite:

