

DAY 1

Keynote Forum



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THE EFFECT OF AN ACRYLIC POLYMER WAX CONTROL ADDITIVE AS A COLD FLOW SOLUTION FOR WAX CHALLENGES

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Maintaining flow in the production and transportation of crude oil is a critical challenge in both on-shore and off-shore environments. Problems such as wax deposition and flow restriction from gelling can occur with changes in temperature and pressure during production, as well as changes in crude oil composition when oil streams are combined. Traditionally, solvent, thermal and mechanical methods have been used as wax remediation treatments. However, chemical treatments are now increasingly employed to mitigate flow assurance problems caused by the naturally occurring paraffins in crude oil. Due to the unique composition of every crude oil, there is no single product capable of treating all flow assurance problems. An acrylic comb polymer has been developed as a dual Pour Point Depressant (PPD) and Wax Inhibitor (WI). A PPD lowers the temperature at which wax crystals form a network which gels and solidifies the oil and a WI reduces the amount of wax which is deposited on pipe walls and other surfaces. This polymer has a strong PPD and/or WI effect in several crude oils yet its mode of action is not fully determined. Cross Polarized Microscopy (CPM) was used to observe the effect of the polymer on wax crystal morphology in several crude oils and to shed light on the mechanism of Pour Point Depression and Wax Inhibition. Early results suggest that the polymer modifies the structure of the wax crystals, making them less angular and less dispersed throughout the oil. It is possible that this reduces the ability of the wax crystals to form a network and gel the oil, or to deposit on metal surfaces. This material also displays a range of other properties and results which makes it an attractive solution to wax challenges in cold flow applications.

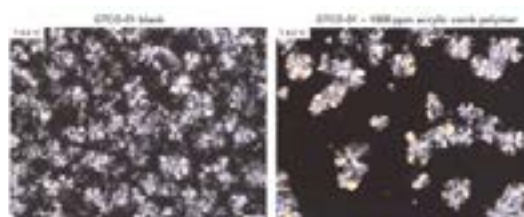


Figure 1: A visual comparison of the wax crystals in an Asian crude oil (with and without polymer). Details of crude oil: Pour Point=39°C, API=33°, Wax Appearance Temperature=54°C. The most common chain lengths in the wax profile are C20 to C35.

Recent Publications

1. Davies C et al. (2013) The development and field application of new surfactant chemistries for applications to heavy oils. Society of Petroleum Engineers. Pages:10.

Biography

Jessica Gould gained her PhD in Synthetic Inorganic Chemistry under the supervision of Professor Martin Schröder at The University of Nottingham. She currently works as a Lead Research Scientist at Croda Europe Ltd specializing in the development of acrylic polymers for a wide range of applications from Personal Care to Battery Additives. Since joining Croda in 2013 she has developed a wide range of expertise in synthesis of new products focusing on tailored dispersants and rheology modifiers.

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ULTRAHIGH-TEMPERATURE PHTHALONITRILE NANOCOMPOSITES WITH FUNCTIONALIZED SILICON-BASED NANOPARTICLES: STRUCTURE, DYNAMICS AND PROPERTIES



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A series of heterocyclic densely cross-linked hybrid nanocomposites were synthesized from bisphenol A-based bisphthalonitrile (BPh) with 0.5wt.% amino-functionalized montmorillonite (MMT) nanolayers, or amino- or epoxy-polyhedral oligomeric silsesquioxane (POSS) nanoparticles. Curing was performed at 260-300°C followed by post-curing at 340-430°C. The reactive nanoparticles were covalently embedded into matrix. FTIR indicated attaining 95% polymerization degree at post-curing and the appearance of phthalocyanine, triazine and isoindoline cycles in the network structure. Nanostructure, dynamics and properties of 0.5 mm thickness films were characterized using scanning transmission electron microscopy (STEM), energy dispersive X-ray spectroscopy (EDXS), FTIR and far-infrared spectroscopy (FIRS), dynamic mechanical analysis (DMA, 0.1-10Hz), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). TGA/DMA/DSC measurements were performed in air or nitrogen mediums. STEM images, EDX spectra and the histograms of Si nanodistribution indicated satisfactory dispersion and uniform distribution of POSS nanoparticles (no nanoclusters) in the matrix. Single MMT nanolayers and 2-3 nanolayers stacks prevailed in the nanocomposites. TGA indicated slight thermal degradation starting from 420-430°C, identically up to 550°C in air and nitrogen mediums. At 550-700°C, TGA curves sharply diverged: thermo-oxidative degradation

in air resulted in dropping mass to char residue of 4-7% whereas 72-74% residual was registered in nitrogen. At low mechanical losses ($\tan \delta \leq 0.03-0.04$), four relaxations were registered for post-cured samples: non- or low-cooperative sub-Tg relaxations at 50-100°C(I) and 200-250°C(II), and $T_g \approx 460^\circ\text{C}$ (III) and 515-540°C(IV); for cured films, $T_g = 380-390^\circ\text{C}$. Transition IV was displaced to 560°C in nitrogen. DMA/DSC showed some suppression of matrix dynamics by nanoparticles: T_g increased by 20-60°C. Glass transition manifested highly cooperative ("quasi-phase transition") behavior with effective activation energy $Q_{act} > 1000 \text{ kJ/mol}$. Dynamic modulus of the nanocomposites $E' \approx (2-3) \text{ GPa}$ decreased by 20-30% at 500°C. After scanning to 570°C in nitrogen medium, no relaxation spectrum and glass transition were observed, and $E' \approx 3.2 \text{ GPa}$ was registered at 20-600°C. The studied nanocomposites have a perspective for use in aerospace, microelectronics, etc.

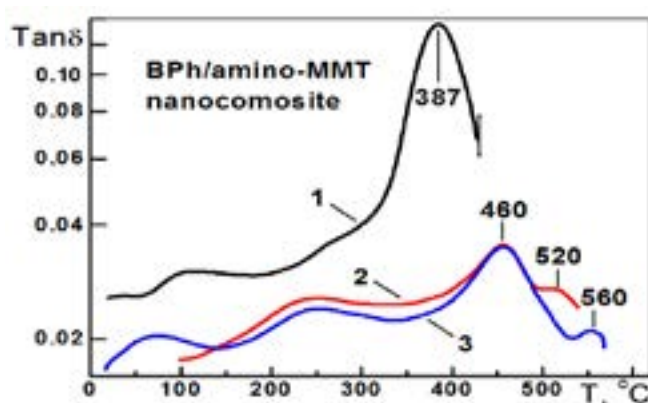
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Figure 1: DMA (1 Hz). Relaxation spectra were obtained for the indicated nanocomposite at heating with the rate of 30C/min in air atmosphere up to 430°C for the sample cured up to 300°C (1) and post-cured up to 430°C (2) and for post-cured sample in nitrogen medium (3).

Biography

Vladimir Bershtein (PhD, 1963, DSc 1980) with his group at Ioffe Institute of the Russian Academy of Sciences has performed numerous experimental studies during a few decades within such polymer physics problems as the nature of relaxation transitions; physics of strength and plasticity of polymeric materials; dynamics and properties of polymer nanocomposites including thermostable (polyimide and cyanate ester-based) and biocompatible ones, and dynamics of polymer hybrids. He is the co-author of numerous publications, e.g., the first book on "DSC of Polymers: Physics, Chemistry, Analysis, Technology", reviews on Far-IR spectroscopy (Adv Pol Sci 114,1994), on the original method of Laser-interferometric creep rate spectroscopy of polymers (Adv Pol Sci 230, 2010), on polyimides and polycyanurate dynamics, etc.

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Recent Publications

1. Bershtein V et al. (2015) The impact of ultra-low amounts of introduced reactive POSS nanoparticles on structure, dynamics and properties of densely cross-linked cyanate ester resins. *Europ. Polym. J.* 67:128-142.
2. Bershtein V et al. (2015) The impact of ultra-low amounts of amino-modified MMT on dynamics and properties of densely cross-linked cyanate ester resins. *Nanoscale Research Lett.* 10:165-179.
3. Bershtein V et al. (2016) Dynamics and properties of high performance amorphous Cyanate Ester-based subnanocomposites with ultralow silica content and quasi-regular structure. *Polymer.* 103:36-40.
4. Bershtein V et al (2016) Silica subnanometer-sized nodes, nanoclusters and aggregates in cyanate ester based networks: structure and properties of hybrid subnanocomposites. *Europ. Polym. J.* 85:375-389.
5. Bershtein V et al. (2016) Thermostable cyanate ester resins and POSS-containing nanocomposites: influence of matrix chemical structure on their properties. *Polym. Adv. Technol.* 27(3):339-349.

DAY 2

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PHENOMENOLOGICAL REVIEW OF LCP/PET DROPLET FIBRILLATION BY REPEATED EXTRUSION FOR NANOFIBER FORMATION

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LCP(Liquid Crystal Polymer) is a high strength polymer which shows characteristics that are the rigid main chain and molecule's arrangement which has directivity. LCP and PET blending is noted in the blend. Because of they have the similar melting temperature and structure. In the LCP and PET blending, the droplet size and dispersion are important point. Therefore understanding of the droplet behavior is very important for the after process. In case of the blended chips supply to make product, once more the chips are extruded from the extruder. So droplet control in the repetitive extrusion is important. But until now, the droplet behavior analysis was not conducted in the repetitive extrusion. Also, droplet behavior change by extrusion number of times was observed that in the process of analysis on blending condition and weight ratio. The droplet behavior change is supposed that relate with flow property, miscibility, surface property of LCP and PET. If nanofiber is manufactured depending on LCP, there is every possibility of utilizing in a higher value-added industry. Although there are some processes to produce nanofiber such as electricity spinning and sea-island type, it still has difficulties that electricity spinning has a low output and sea-island type is restricted to reduce fiber diameter. It will be effective to solve the existing problems as mentioned above that if material of droplet shape is able to become consecutive fiber morphology through stretching process. The research that deal with making continuity through the way to regulate size of droplet has not yet been achieved in existing dissertations of manufacturing of fibers related to droplet stretching method. This study is planned to verify control of droplet via study of its behaviors that are influenced by repetitive extrude LCP and PET blend substance and confirms size changes of droplet while it is extrude repeatedly. These changes show size growth according to increase number of extrusion and changes of droplet's location are checked as well. Distributions of droplets were observed to LCP and

PET blending process for conjugate spinning. Droplets were distributed relatively evenly in the initial extrusion process. But the secondary and third the size of the droplet was increased and the phenomenon was founded that the droplet was gathered in the center. This phenomenon was assumed that the miscibility of LCP/PET and the flow characteristics correlate with the phenomenon, so conducted the analysis. In this study, to analyze distribution and component of droplet was conducted. Also miscibility of LCP/PET was analyzed.



Figure 1: Test equipment for LCP/PET droplet behavior by repeated extrusion

Recent Publications

1. C.H. Song and A.I. Isayev, LCP droplet deformation in fiber spinning of self-reinforced composites [J], Journal of Polymer, 2001, 42(6) : 2611-2619
2. W.N. Kim and M.M. Denn, Properties of blends of a thermotropic liquid crystalline polymer with a flexible polymer(Vectra/PET) [J], Journal of Rheology, 1992, 36, 1477-1498
3. W.G. PERKINS, The Effect of Blending Temperature, Composition, and Shear Rate on PET/Vectra A900 LCP Blend Viscosity and Morphology [J], Journal of Applied Polymer Science, 43, 329-349

4. P. MAGAGNINI, On the Use of PET-LCP Copolymers as Compatibilizers for PET/LCP Blends [J], *Journal of Polymer Engineering and Science*, 1996, 36(9), 1244-1255
5. K. NAKAYAMA, Structure formation and miscibility of sheets from PBT and LCP blends [J], *Journal of Materials Science*, 2001, 36, 3207-3213

Biography

Deanna Prof. Han-Yong Jeon, geosynthetics/technical organic materials researcher and he was the 32nd President of Korean Fiber Society (2014~2015). He has published more than 845 proceedings in domestic and international conferences. He wrote 20 texts including 'GEOSYNTHETICS' and also published 143 papers in domestic & international journals. He has awards of Marquis Who'sWho - Science and Engineering in 2003~2017 and also, he got the 33rd Academy Award of Korean Fiber Society in 2006 and "Excellent Paper Award of 2012" by The Korean Federation of Science and Technology Societies.

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