



Symposium 11

Processing of Composites and Filled Systems



STUDYING ON PREPARATION AND REINFORCEMENT MECHANISM OF HIGH PERFORMANCE MULTI-WALLED CARBON NANOTUBE/ULTRA-HIGH MOLECULAR WEIGHT POLYETHYLENE COMPOSITE FIBERS

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Ultrahigh molecular weight polyethylene (UHMWPE) has the simplest structure and in its fully extended form can have tensile modulus and tensile strength approaching theoretical estimation of ~200GPa and 19GPa respectively. However, there is a big gap between the mechanical properties of the current commercial PE fiber and the theoretical values [1-2]. In this study 5wt% acid-treated multi-walled carbon nanotube (MWCNT) have been adopted as the fillers and successfully developed a gel-spinning process using twins-extruder for fabricating highly oriented MWCNT/UHMWPE composite fibers. The modulus and the strength of the fibers enhance to 137 GPa and 4.2 GPa respectively, which are the best specific mechanical properties among the current commercial fibers. An extensive experimental study was carried out to characterize the morphology and reinforcement mechanism of the highly oriented composite fibers. The mechanism of reinforcement has been studied using a combination of high resolution scanning electron microscopy and micro-Raman spectroscopy. Carbon nanotubes alignment along the tensile draw direction has been observed at high elongation ratios. Such alignment induces strong interfacial load transfer both at small and large strains to enhance the stiffness and tensile strength of the composite fiber. The detailed morphology of the fiber interface is studied using transmission electron microscopy. A direct observation of interface between the matrix and CNTs benefit our deeper understanding of the reinforcement mechanism. [1]Pennings A.J., Smook J., de Boer J., Gogolewski S., van Hutten P.F., Process of preparation of ultra-high strength polyethylene fibers, *Pure Appl. Chem.*, 55, 777-798, 1983 [2]Matsuo M., Sawatari C., Elastic modulus of polyethylene in the crystal chain direction as measured by x-ray diffraction, *Macromolecules*, 19, 2036-2040, 1986

S11-460

Development and characterization of electrically conductive polymeric based blends for PEMFC bipolar plates

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The main objective of this research was to study the synergistic effects of adding different electrically conductive fillers to a polymer matrix in order to optimize blend conductivity and its processability by twin-screw extrusion in final bipolar plate (BPP) shape. The selected polymer matrix was a low viscosity Polyethylene Terephthalate (PET). The conductive fillers were composed of a high specific surface area Carbon Black (CB) and synthetic flake graphite (GR). A third conductive additive, consisting of Silver Coated Glass particles (SCG) or multi-walled Carbon NanoTubes (MWCNT), was also added at a low concentration in order to study its synergistic effect on blend electrical conductivity. As the developed blends have to meet properties suitable for Proton Exchange Membrane Fuel Cell (PEMFC) BPP applications, they were characterized for their electrical resistivity, mechanical properties, and oxygen permeability. Blend morphology was characterized by Scanning Electron Microscope (SEM) in order to elucidate its effect on through-plane electrical conductivity. The influence of PET crystallinity on the electrical conductivity was also studied on three PET/CB blends cooled at three different cooling rates. It was observed that higher electrical conductivity was obtained with blends of higher crystallinity. During PET crystallization, CB particles are expelled from the crystalline regions, leading to a more developed conductive network in the interspherulics and in the amorphous regions. Through-plane electrical resistivity of about 0.3 Ω.cm, and oxygen permeation rate of 3.5×10⁻⁸ cc.cm⁻².s⁻¹ were obtained for only 30wt% of conductive fillers.



SELF-DIAGNOSIS COMPOSITE MATERIAL PROPERTIES FOR CIVIL APPLICATION

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At present much attention is devoted to the realization of innovative materials to increase the safety of civil structures, the so-called “self-monitoring materials”, that behave contemporarily as mechanical reinforcement and sensor of the structure in which they are inserted [1,2]. More recently a new trend [3,4] suggests the use of carbon nanocomposites for the realization of multifunctional materials, since they seem to offer excellent electrical properties, that are essential for achieving the required monitoring performance. The present research is focused on the design and manufacture of two typology of hybrid self-sensing nanocomposite rods, consisting on a coaxial structure with an internal conductive rod covered with glass fibres. The electrical conductive phase is made either using carbon nanocarbon particles (CNPs) or carbon nanofiber (CNFs). In both cases epoxy resin is the dispersive medium. The self-monitoring performance of the so-realized materials were tested by monotonic tensile tests, during which the electrical resistance variation of the conductive part was acquired and correlated to the mechanical load. The effect of particular filler on self-diagnosis properties was then evaluated. The results showed that in both cases self-monitoring properties were achieved, even if the carbon nanoparticles system reached higher sensitivity and reliability than in the case of carbon-nanofibres. This result has to be directly correlated to the filler dispersion, that is much more complex in the case of nanofibres. References 1.F.Nanni, G. Ruscito, G.Forte, G.Gusmano, *Smart Materials and Structures*, Vol.16 (2007) pp.2368- 2374 2.Bakis C.E., Nanni A, Terosky J.A. and Koehler S W, *Composite science and technology*, Vol. 61 (2001) pp. 815-823 3.Y. Okuhara, H. Matsubara, *Carbon* Vol 45 (2007) pp. 1152-1159 4.Joung-Man Park, Dae-Sik Kim, Sung-Ju Kim, Pyung-Gee Kim, Dong-Jin Yoon, K. Lawr

S11-1017

New Models for Fiber Orientation in Injection-Molded Composites

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Fiber orientation models based on Jeffery's equation, modified with an isotropic rotary diffusion term by Folgar and Tucker, have become a standard tool for predicting fiber orientation in injection molding. However, recent experiments show that this model seriously under-predict the width of the randomly-oriented core in parts with short to medium flow lengths. Additionally, in long-fiber injection-molding materials the shell layers exhibit high levels of cross-flow alignment but very low out-of-plane alignment, an orientation state that cannot be reproduced by standard model. To address these problems we present two major improvements to the standard fiber orientation model. The problem of thick cores is addressed by slowing the kinetics of alignment. The new model is developed by deriving rate equations for the eigenvalues and eigenvectors of the orientation tensor, reducing the eigenvalue growth rates by an empirical constant, and re-assembling the equations. The resulting model, called RSC, is objective, and behaves very much like the standard model but with slower kinetics. For long-fiber thermoplastics we modify the rotary diffusion term to introduce anisotropic rotary diffusion (ARD). The scalar interaction coefficient C_I is replaced by a tensor C , and the corresponding rate equation for the orientation tensor A is re-derived, correcting an error in the model of Fan and Phan-Thien. The new rotary diffusion tensor C is a function of orientation A and rate of deformation D , and we demonstrate a systematic procedure for fitting the model parameters to experimental data. Both models are compared to experimental data from simple injection-molded parts, and show greatly improved agreement with data compared to the standard model. The RSC model is also compared to rheological data, and accurately predicts the shear stress in shear transient reversal.



An innovative method for the characterization of the nanoclay dispersion in rubber compounds and blends

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The online electrical conductance has been used as a novel method to characterize the dispersion of nanoclay in rubber compounds and blends. A synchronized increase of the online conductance measured directly in the mixing chamber of the internal mixer along with the dispersion of nanoclay in the rubber matrix was found. The conductivity signal is sensitive to the intercalation/exfoliation process of nanoclay in rubber compounds. The correlation between the online conductance and the development of the nanoclay dispersion/intercalation/exfoliation determined by various experimental techniques like AFM, SAXS, bound rubber measurement, and surface tension measurement as well as tensile testing has been investigated. It has been observed that the technological parameters strongly influence the behavior of the nanoclay dispersion. Furthermore, the method has been used successfully to investigate the dispersion of nanoclay in immiscible NBR/NR blends. The nanoclay not only has the tendency for specific phase distribution but also strongly effects the morphology of the specific phase. Based on the online conductivity, a deeper insight into the mixing kinetics and phase specific clay transfer as well as blend morphology development was achieved.

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Effect of organoclay modification on the mechanical, morphology, and thermal properties of injection molded polyamide 6/polypropylene/montmorillonite nanocomposites

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Polyamide 6/polypropylene (PA6/PP = 70/30 parts) blends containing 4 phr (parts per hundred resin) of organophilic montmorillonite (OMMT) were prepared by melt compounding using co-rotating twin-screw extruder followed by injection molding. The effect of organoclay modification on the mechanical and morphological properties of nanocomposites were investigated by using x-ray diffraction (XRD), transmission electron microscopy (TEM), tensile, flexural, and impact tests. The thermal stability and thermal properties of nanocomposites was characterized by using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The morphology and properties of the PA6/PP nanocomposites were determined by the alkyl ammonium salt length, that is, the hydrophobicity of the organic modifier. XRD and TEM revealed the formation of exfoliated structure for the nanocomposites prepared using stearylamine modified montmorillonite and commercial organoclay. On the contrary, a mixture of intercalated and exfoliated structures was observed for the nanocomposites prepared using 12-aminolauric acid and dodecylamine modified montmorillonite. Incorporation of OMMT into PA6/PP matrix increased stiffness and strength but at the expense of ductility. The nanocomposite prepared using stearylamine modified montmorillonite displayed the best mechanical properties and thermal stability. This was attributed to better exfoliated structure in the nanocomposites.



DEFORMATION, FRACTURE, AND TRIBOLOGICAL BEHAVIOR OF MoS₂ FILLED HMWPE COMPOSITES

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High molecular weight polyethylene (HMWPE) and its composites are widely used as bearing components, gears, guide rails, in food treatment and medical equipment because of their excellent friction and wear characteristics, corrosion resistance and mechanical properties. It is widely known that the tribological behavior of polymers can sometimes be greatly improved by filling them with inorganic particulate compounds, but their effect on mechanical properties is not well established so far. Through this work molybdenum bisulphide (MoS₂) HMWPE based composites were prepared, and the effect of filler on the stress-strain, fracture and tribological behavior of composites was studied. MoS₂ powder was compounded with a commercial HMWPE powder by ball milling. Afterwards, the mixture were filled into a mould and pressed into plaques in a hydraulic press followed by hot pressing. Plaques were allowed to cool slowly in order to avoid thermal residual stresses. Four compositions were prepared – 5, 10, 20 and 30% – in order to elucidate the optimum proportion of the solid lubricant in HMWPE for minimum wear rate. Abrasion of samples was carried out using the Dry Sand/Rubber Wheel Technique. The influence of blending and composition on the wear rate was evaluated. Stress- strain behavior and fracture toughness of materials was characterized under quasistatic conditions (at 10 mm/min) using dog-bone and single edge notched SE(B) specimens respectively. Also the energy release rate, GIC, of composites was determined at 1 m/s. Wear rate was diminished and wear mechanisms were changed by the addition of the solid lubricant. It was found that the optimum proportion of MoS₂ in HMWPE for minimum wear rate is 10%. However, fracture toughness of HMWPE is clearly deteriorated and also the fracture behavior changed from ductile to brittle with the addition of MoS₂.

S11-295

Processing – Property Relationship of Long Fiber Reinforced Polypropylene Composites

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Long glass fiber reinforced thermoplastics (LFRT) are in great demand in the compounding industry, with a global market size of 108 million kg (239 million lbs) and a value of \$450 million, reports U.S based consultancy Principia Partners. This study describes the development of Long Fiber Reinforced Polypropylene (LFPP) composites and the effect of fiber length, compatibilizer content on its mechanical properties. LFPP pellets of different sizes were prepared by extrusion process using a specially designed impregnation die and these pellets were injection molded to develop LFPP composites. Maleic-anhydride grafted polypropylene (MA-g-PP) was chosen as a compatibilizer and its content was optimized by fiber pullout test. Critical fiber length was calculated using interfacial shear strength. Fiber length distributions were analyzed using profile projector and image analyzer software system. Fiber aspect ratio of more than 100 was achieved after injection molding. The results of the tensile and flexural properties of injection molded long glass fiber reinforced polypropylene with a glass fiber volume fraction of 0.18 are determined. It was found that the difference in pellet sizes improves the mechanical properties by 3-8%. The improvements in properties are significant in case of compatibilized LFPP composites. Efforts are made to theoretically predict the tensile strength and modulus using the Kelly Tyson and Halpin Tsai model respectively. Notched izod impact property was studied for LFPP composites prepared with and without compatibilizer for different pellet sizes. Failure mechanism due to sudden impact was analyzed with scanning electron micrographs pictures and was correlated with impact property of LFPP composites. Fracture and failure behaviour of injection molded LFPP composite were studied and relationship between fracture toughness and microstructure of LFPP composite was analyzed. The microstructure of the composites was characterized by the dimensionles



The processing of optically transparent polystyrene blends filled with cross-linked polystyrene beads.

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In order to assist understanding of both processing and properties of polymer blends, a series of polymer blends have been made where a polystyrene matrix has been blended with spherical crosslinked polystyrene particles. The blend has sufficient transparency that optical observations can be made during processing. Different concentrations of blends were prepared by one of two routes; namely mechanical blending and blending using solvent mixing. Rheological data was obtained using standard viscoelastic rheometers and in addition processing data was obtained using a Cambridge Multipass Rheometer MPR[1]. Rheological data and optical observations of the blends were carried out using the MPR for an entry/exit slit flow[2] and an extensional flow cross-slot configuration[3]; using both flow birefringence and direct optical visualisation. The MPR pressure profile processing data showed differences between the blends that were prepared by mechanical and solvent processing and the pressure change with volume fraction and flow-rate has been modelled in relation to the differences in hydrodynamic volume of the cross linked beads within the matrix for the solvent and mechanically blended materials. The birefringence and direct visualisation observations provided insight into the similarities and differences in processing behaviour of the matrix alone and the blended materials. In addition an unexpected cavitation phenomenon was observed for certain processing conditions of the blend. References [1] Mackley M.R., Marshall R.T.J., Smeulders J.B.A.F., "The multipass rheometer", *Journal of Rheology*, 39(6), 1293-1309 (1995). [2] Collis M.W., Mackley M.R., "The melt processing of monodisperse and polydisperse polystyrene melts within a slit entry and exit flow", *J. Non-Newtonian Fluid Mech*, 128, 29-41 (2005). [3] Coventry K.D., Mackley M.R., "Cross-Slot Extensional Flow Birefringence Observations of Polymer Melts Using a Multi-Pass Rheometer", Submitted to *Journal of Rheology* 2007

S11-344

Product Development with Thermal Conductive Polymers and the Need for Integrated Use of Simulation

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There is a growing need for innovative concepts in the thermal management of technical applications due to increasing device complexity (electronification), constraints on construction method (miniaturization) and altered thermal framework conditions. An efficient approach is to take existing components and generate further functions. For example, plastic housings could take on thermal functions in addition to mechanical ones. Thermal conductivities up to 20 W/(mK) for plastics compounds can be obtained by adding metallic and ceramic fillers. The high filler fraction and the associated thermal conductivity lead to changes in process conditions, relative to standard injection molding. Anisotropic fillers are being oriented during the injection molding process. Dependent on the flow conditions, different anisotropic orientation and a distribution of conductivity values are obtained. By changing process settings such as mold temperature or injection speed the flow conditions and hence the filler orientations are being changed. This leads to a process induced distribution of thermal conductivity. Effective product development with thermal conductive polymers requires an overall designing concept. Integrated product development is defined by three basic columns: material properties, processing and product design. Only by concurrent operations connecting these columns a successful product development can be realized. Thereby simulation provides the necessary means for the needed interface information. In this paper experimental results show the influence of processing parameters on thermal and mechanical part properties. These results point out the necessity for a connection between process and product simulation when designing injection molded plastic parts out of thermal conductive polymers. Furtheron the potential of simulation as approach to analyze the injection molding process of thermal conductive polymers as well as the prediction of part properties is presented.



The influence of reactive oligomers on processing behaviour and properties of flame retarded Polypropylene/Magnesiumhydroxide-composites

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To render polypropylene effectively flame retarded a minimal filler loading of 55 wt% of magnesiumhydroxide (MH) is usually required. This results in a drastic deterioration of the processability and mechanical properties of the composites. State-of-the-art surface treatment of MH with long chain carboxylic acids or silanes, can increase processability and certain mechanical properties of the composites but on the other hand flame retardancy can be affected as well. In our study we investigated novel reactive oligomers (RO) as surface modifiers for magnesiumhydroxide and their effect on processability, filler dispersion, mechanical properties and flame retardancy of PP-based composites. The RO's consist of different blocks: maleic anhydride containing groups provide a link to the mineral filler surface, reactive blocks containing peroxide moieties will radically react and can graft onto the surrounding matrix or act as starting points for graft polymerisation of i.e. butylacrylate to form a compatibilising polymer shell around the filler particles. Functional blocks control the reactivity of the RO's as well as they determine properties such as surface energy of the particles coated with them. We found that the RO's led to the formation of an interphase layer between filler particles and host polymer that resulted in improved mechanical performance especially when considering the stiffness-toughness-balance of the composites. At the same time the processability of the composites was maintained or even improved despite the high filler loading of 60 wt%. In contrast to composites containing standard modified magnesiumhydroxide the flame retardancy of the RO-based composites was maintained or even improved thus exhibiting a good balance between physical properties and flame retardancy. This work was supported by FLARETPOl-project, which is co-funded by the Sixth Framework Programme of European Commission (NMP3-CT-2005-516998).

S11-601

PROCESSING AND CHARACTERIZATION OF ELECTRICALLY CONDUCTIVE IN-SITU MICROFIBRILLAR POLYMER COMPOSITES

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Electrically conductive in-situ microfibrillar polymer composites were prepared by extrusion and hot stretching the poly(ethylene terephthalate) (PET), multiwalled carbon nanotube (CNT) and high density polyethylene (HDPE). Extrusion process conditions, hot-stretching speed and CNT content (0.5 wt. %) in the composites were kept constant throughout the experiments, whereas HDPE/PET ratio in the samples and molding temperature were changed. The produced composites were characterized in terms of their morphology, electrical, mechanical and thermal properties. Surface energy measurements and scanning electron microscopy (SEM) analysis showed that CNT was preferentially located in PET phase. It was seen from micrographs of the hot-stretched composites that in-situ PET-CNT microfibrillar structure formed in HDPE phase. Electrical conductivities of the in-situ microfibrillar composites were in semi-conductor range at 0.5 wt. % CNT content for all HDPE/PET ratios. The increase in the molding temperature of hot-stretched composites decreased the electrical conductivity, due to the deterioration of the PET-CNT microfibrillar network. Microfibrillar reinforcement improved the tensile and impact strength of the composites when compared with those of conventional composites. The crystallinity of PET phase increased as a result of the nucleation effect of nano sized CNT particles.



POLYOLEFINS/CELLULOSE FIBRES COMPOSITES : DISPERSION, COLORATION, COMPATIBILIZATION AND NANOCOMPOSITE ELABORATION.

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Low Density Polyethylene (LDPE)/cellulose fibres composites have been processed by high shear extrusion with water injection to help dispersion of fibres. Optimization of extrusion parameters is necessary to reach a fine dispersion of the filler, limiting the presence of aggregates. Injection of water during extrusion improves the dispersion but the main effect is a spectacular decrease of the discoloration (yellowing) due to cellulose degradation [1]. Young modulus increases with cellulose content and reinforcing effect is more important above 10% by weight. Due to the weakness of the interface, ductility is reduced compared to LDPE. Compatibilizing agents like polyolefins grafted on maleic anhydride and ionomers have been used to enhance the adhesion between the matrix and the filler. The mechanical properties of the samples have been evaluated. Processing cellulose fibres based nanocomposites needs to treat the filler before blending it with a polymer. Homogenization process has been used to release microfibrils from cellulose fibres. Then the homogenized cellulose has been freeze-dried and blended with LDPE. High shear extrusion with water injection under pressure was used to elaborate the nanocomposites. However large cellulose aggregates remained after the extrusion due to the irreversible reaggregation of the cellulose microfibrils during the freeze-drying step [2]. The homogenized cellulose had thus to be treated with a surfactant in order to avoid this reaggregation and allow a fine dispersion in the melt.[1] J. Soulestin, N. Quiévy, M. Sclavons, J. Devaux, Polyolefins-Biofibre Composites : A new way for an industrial production, Polymer Engineering and Science, 47, 4, 467-476, (2007). [2] L. Heux, C. Bonini, US 6,967,027 B1, November 2005.

S11-753

Characterization of flame retardancy and effect of synergism on PET based composites via phosphate based flame retardant additives

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Flame retardancy of poly(ethylene terephthalate), (PET) was improved with the usage of phosphate based flame retardants. Crystalline PET was used as matrix material and flame retardancy effects of different amounts and combinations of triphenyl phosphate, triphenyl phosphine oxide, and microwave produced boron phosphate were examined. Flame retardant additives were used up to 20 weight percent of the matrix polymer. Composites were prepared by using a twin screw extruder and injection molded for characterization purposes. They were characterized in terms of both flame retardancy behavior and mechanical properties. Flame retardancy of the composites were determined by conducting limiting oxygen index (LOI) test and horizontal burning rate test. Another important property, which is smoke emission during fire, was also evaluated in terms of percent light transmittance. According to the LOI test, LOI of neat PET was increased from 21% up to 36% with the addition of flame retardant additives. Regarding the analysis, boron phosphate was determined as a good smoke suppressant for the PET matrix. Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) were performed to examine further degradation of the polymer matrix. In addition to flammability properties, tensile and impact properties of the composites were also improved with the addition of flame retardant additives. Addition of boron phosphate, and triphenyl phosphate increased the tensile strength and the impact strength of the composites, respectively, when compared with neat PET.



Processed biopolymer films filled with modified Montmorillonite for food packaging applications

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During the last decade, the use of clays for polymer reinforcement has attracted a lot of attention since it is possible to get an enhancement in mechanical, thermal, barrier and flammability properties compared to matrix materials. In packaging, huge amounts of plastics materials are used, and therefore efforts are focused towards biodegradable/biocompatible polymers. Some biopolymers have already an application in food packaging industry but these applications would be increased if materials were reinforced. One approach to such improvement is the use of nanofillers in bioplastics leading to biopolymer nanocomposites. The use of modified nanoclays for this purpose has been reported previously[1]. In this work, different ammonium salts suitable for food contact applications are used as modifiers to get hydrophobically modified montmorillonite, and hence to obtain better compatibility between the biopolymer and the filler (nanoclay). Different methods have been used to obtain the modified montmorillonite[1-2]. The obtained nanofillers have been characterized by FTIR, X-ray diffraction, and TGA, in order to study the composition, structure and thermal stability. The fillers have been used to reinforce polylactic acid, among other biopolymers in a microextruder (DSM Xplore), to obtain thin films, which have been characterized using different techniques such as FTIR, X-ray diffraction, DSC and TGA to evaluate composition, structure and thermal stability. Results have been compared with bulk films, and other films reinforced with commercial fillers. 1.Salas-Vicente, J., et al., Aminoacid modified montmorillonite as reinforcement in polyhydroxybutyrate matrices. 1st International Conference on Biodegradable Polymers and Sustainable Composites, 2007: p. P2-11.2.Rajkiran R Tiwari, et al. Synthesis and characterization of novel organo-montmorillonites. Applied Clay Science, 2007. In press.

S11-824

Investigation on Poly(butylene terephthalate) (PBT) nanocomposites prepared with Bis(hydroxyethyl terephthalate) (BHET) modified clay.

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Polymer-layered silicate (PLS) nanocomposites have attracted much attention recently because of their remarkable improvements in comparison to the neat matrix, such as thermal, mechanical and barrier properties. In order to render layered silicates miscible with other polymeric matrices and promote an exfoliated structure, normally hydrophilic silicate surface has to be converted in an organophilic one. Generally, this can be done by ion-exchange reaction with cationic surfactants such as alkylammonium cations. Since the processing temperature of some polymers like Poly(butylene terephthalate) is higher than the thermal stability of these organoclays, much attention has been directed toward the development of improved organophilic treatments for layered silicates. In this study an innovative organoclay with a better thermal stability based on the Bis(hydroxyethyl terephthalate) (BHET) intercalated in the montmorillonite (MMT) galleries has been proposed. The interlayer distance of MMT is affected by the amount of BHET, with a maximum of 21.3 Å occurring in correspondence of 50% wt of modifier, whilst Na⁺-montmorillonite has a d-spacing of 11.7 Å. The effect of the BHET condensation and its resulting molecular weight on the subsequent clay exfoliation/intercalation during PBT/nanocomposites preparation via melt compounding has been investigated. The morphological and thermo-mechanical properties were studied using wide angle X-ray diffraction and transmission electron microscopy, TGA, DSC and DMA.



PP-Clay Nanocomposites: effect of short-chain additives on rheology and mechanical properties

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The influence of low molecular weight additives containing polar groups and modified, polyolefin-based compatibilisers on PP-clay nanocomposites (PPCN) has been studied, in terms of intercalation and exfoliation achievable by melt-state mixing processes. X-ray diffraction (XRD) analysis shows that the interlayer spacing of montmorillonite clay increases dramatically, whilst transmission electron microscopy (TEM) shows increased particle dispersion, when short chain, organic additives (typically amide-type) are included. Contact angle / surface energy data has confirmed the migration of these additives into the clay galleries. A mechanism for co-intercalation is proposed in terms of hydrogen bonding between the additives and the silicate layers. Shear flow data have been used to interpret the mechanism of intercalation observed during melt-state mixing processes and improved thermal stability (relative to unfilled PP) has been achieved. Modified melt elasticity is obtained with the PPCN's leading to reduced die swell characteristics in extrusion. In addition, the low strain modulus of PPCN is much improved, relative to unfilled PP, whilst the filled compounds retained a similar level of yield strength in tension. Mechanical test data for PPCN have been interpreted in terms of the degree of exfoliation, additive content and differences in PP crystallinity. Keywords: mixing / extrusion / PP nanocomposites / exfoliation / rheology / mechanical properties

S11-879

Fully Absorbable Orthopedic Composites via a Modified Pultrusion Process

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Of importance to orthopedic procedures are repair materials that can carry significant loads without excessive deformation. In addition, these materials need to be biocompatible, bioabsorbable, and conducive the rapid replacement of the repair material with native bone. To meet these requirements, we have designed composites using two biocompatible/bioabsorbable polymers, poly(L-lactic acid) (PLLA) and poly(ϵ -caprolactone) (PCL), with the latter filled in a carefully designed fashion with nano needles of hydroxyapatite (HA). Discussed in this talk are the processing challenges and solutions for achieving a flex modulus of 8 to 10 GPa, with particular emphasis on a pultrusion process. The feed to the pultrusion die is a stabilized suspension of the HA needles (7:1 aspect ratio) in a solution of PCL, whereas the fiber "bundle" is a single yarn of PLLA fiber. This small scale is needed to maximize the content of HA in the final composite. Variables studied include the ratio of HA to PCL in the suspension, the solids content of the suspension and various geometrical variables in the die design that influence the shear-rate profile along the fiber path. The goal of the pultrusion step is to coat all the fibers with a matrix of PCL reinforced with highly aligned HA needles.



Development and Properties of a Microfibril Reinforced Composite Container from a PE/PET Blend

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Polymer-reinforced polymer composites have been successfully created from blends of engineering and commodity plastics. These materials contain molecularly oriented in-situ microfibrils evenly dispersed throughout a homogeneous matrix and are manufactured using an extrusion, fibrillation and matrix consolidation process. This paper investigates several key aspects of the manufacturing process and their influence on the development of the polyethylene/poly(ethylene terephthalate) (PE/PET) composite at various stages of production. Comparisons are made between the mechanical properties achieved for composites created via compression moulding and those made via injection moulding. Scanning electron microscopy (SEM) is used to understand the evolution of the dispersed PET as it is extruded and to assess the effects of die dimensions on average microfibre diameter in the extrudate. Oxygen permeabilities are correlated with the tensile properties of various composite films. Finally, microfibril reinforced containers are produced via injection moulding and the mechanical and barrier properties of these are compared to standard PE vessels.

S11-964

PRODUCTION OF MODIFIED CLAYS AND THEIR USE IN POLYPROPYLENE MATRIX NANOCOMPOSITES

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Effects of one phosphonium salt and three alkylammonium salts on clay modification and the properties of binary nanocomposites composed of polypropylene and the modified clays were investigated. One type of clay used was Na⁺-bentonite rock mined from Tokat/Resadiye, while the other clay was pure grade montmorillonite. Use of three types of alkyl ammonium salts and one phosphonium salt allowed comparison between the effects of phosphonium and ammonium salts on clay modification, while effects of alkyl chain length could be observed by comparing the three ammonium salts. Nanocomposites with different bentonite compositions (% 2-4) were produced in order to see the effects of bentonite concentration on the mechanical properties of the samples. All samples were prepared by a corotating twin screw extruder, followed by injection molding. XRD and SEM analysis were done for characterization of modified and unmodified clays as well as the morphology of the nanocomposite samples. Mechanical properties of the nanocomposites were evaluated by tensile and flexural tests. Thermal properties of the modified clays and the nanocomposites were observed by Differential Scanning Calorimetry (DSC) and Thermal Gravimetric Analysis (TGA). Rheological behaviour of the nanocomposites was evaluated by Melt Flow Indexer (MFI) and Mechanical Spectrometer. X-Ray results showed that the d-spacing of bentonite increased from 11.99 Å to 17.87 Å following modification with hexadecyltrimethylammonium bromide (HDTMA). The mechanical properties also showed improvement in nanocomposites prepared with modified clays in comparison to nanocomposites prepared with untreated clays.



High Flow PP Nanocomposites - Processing and Properties

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Recent development in clay/nanofiller chemistry and better mixing techniques enable industries to make polyolefin based nanocomposites having superior performance compared to conventional materials. Nano-clay Compounding using deep-flighted twin-screw extruders is a promising approach to prepare PP nanocomposites for molding applications need improved flexural modulus without decrease in impact strength and increase in specific gravity. Improvement in mechanical properties of PP nanocomposites, not only depends upon resin characteristics and compatibilizer, but also greatly depends upon the characteristics of the mixing elements in the extruder. In the present study PP grades with MFI 1.5 gm/10min to 40 gm/10min were used and corresponding changes in processing behavior to achieve maximum exfoliation are presented. X-RD and SEM was carried out to study clay dispersion and mechanical properties using ASTM standards. Results discuss the selection of extruder configuration, useful processing parameter to achieve maximum exfoliation in high flow PP grades and performance properties of these nanocomposites. It was observed that upto 70% increment in modulus can be achieved in high flow PP grades without sacrificing the impact strength.

S11-1083

Polymer-Graphite Nanocomposites: Effective Dispersion and Major Mechanical Property and Conductivity Enhancements via Solid-State Shear Pulverization

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The first well-exfoliated and well-dispersed polymer graphite nanocomposites made using only untreated, as-received graphite and polymer was prepared by a novel, continuous process called solid-state shear pulverization (SSSP). X-ray diffraction of 2.5 wt% graphite-polypropylene (PP) nanocomposites made via SSSP revealed an almost total suppression of the scattering peak characteristic of the inter-graphene sheet spacing (0.335 nm) within graphite; electron microscopy revealed the presence of nanoplatelets in the nanocomposite with thicknesses of several to thirty layered graphene sheets. Rheological measurements of the nanocomposites made by also revealed the solid-like character associated with a well-dispersed/exfoliate state. In contrast, PP-graphite hybrids made by melt extrusion had agglomerates with length scales up to hundreds of micrometers. Relative to neat PP, 2.5 wt% graphite-PP nanocomposites exhibited a 100% increase in room-temperature Young's modulus and a 60% increase in yield strength. Such property enhancements have not been previously observed in polymer-graphite nanocomposites/hybrids of the same or lower graphite content (treated or untreated) and produced without solvent/sonication or in nanocomposites made by co-processing PP and organoclay leading to confirmed exfoliation. Dramatic increases in PP crystallization rate and electrical conductivity were also observed in the nanocomposites relative to neat PP. The effects of graphite content and pulverization parameters on the graphite dispersion and enhancement of properties will also be discussed.



Effects of Spherical Nanoparticles on the Impact Strength and Fracture Mechanisms of Melt Compounded Polycarbonate/Alumina Nanocomposites

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In the previous study, it was shown that spherical nanoparticle dispersion within a polymer matrix can be facilitated using the melt compounding method by coating the nanoparticles with a thin layer of polymer. This paper presents the effects of spherical alumina (Al₂O₃) inclusions of various sizes and loading contents on the mechanical properties and fracture mechanisms of polycarbonate (PC) nanocomposites. The PC matrix was compounded with both untreated and poly(styrene–maleic anhydride) (SMA) copolymer-treated alumina nanoparticles using a high intensity thermokinetic mixer (K-mixer). Field emission scanning electron microscopy (FESEM) was used to investigate the fracture surface of the PC/alumina nanocomposites and the PC neat resin. Further chemical analyses were performed to verify the covalent bonding between the copolymer coating and the alumina using Fourier transform infrared spectroscopy (FTIR) and electron spectroscopy for chemical analyses (ESCA). In addition, gel permeation chromatography (GPC) was employed to determine the molecular weight of the unprocessed and processed PC neat resin to evaluate the extent of degradation induced by the thermokinetic mixer. It was found that the alumina nanoparticles were capable of improving the impact strength of the PC/alumina nanocomposites through microcraze formation. In addition, it was found that there was an optimum nanoparticle size and loading content leading to the best impact strength.

S11-1135

Thermal and Dynamic Mechanical Properties of Polypropylene Reinforced with Sugarcane Fibers

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Natural fiber composites slowly take root mainly in automotive industries, thus, different natural fibers have been widely studied. In this work, the effect of sugarcane bagasse and straw fibers on the thermal and dynamic mechanical properties of the polypropylene has been investigated. Both pretreatment with hot water and NaOH pulping were performed for fiber performance and matrix interaction improvements. Neither chemical modification of fibers and matrix nor additives were applied in these composites processing. Otherwise, the possibility for use of fibers without hard and expensive modification methods could be an excellent alternative for replacement of synthetic fibers as reinforcement in thermoplastic composites. The study of composites reinforced with natural fibers has been carried out by means of differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA). The results have shown that both fibers behave as nucleant agents for the crystallization of polypropylene. A noticeable increase of the PP crystallization temperature in the presence of both fibers has been observed. This effect is more sensible with cellulose and pretreated fibers. In addition, the percentage of PP crystallinity (X_c) increase in the presence of raw sugarcane bagasse and straw and hardly varies with the amount of fibers. The fibers give rise to a sensible increase of the polypropylene stiffness (storage modulus), whereas a slight decrease of the damping factor (tan delta) has been observed. Furthermore, the PP glass transition temperature (T_g) decreases in the presence of the natural fibers. The activation energies for the relaxation process increase with the incorporation of both fibers. As a conclusion, the crystallinity and dynamic mechanical properties increases could show that the simple treatments on natural fibers causes a meaningful interface between fiber and matrix, improving its composites performance. Acknowledgements to FAPESP, FCT and CNPq.



Influence of clay and organoclay addition on the tensile properties and thermal aging of PP/sisal composites

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The effect of pristine and organoclay addition on the properties of PP/sisal composites as a function of processing conditions was investigated. A local pristine clay was chemically modified (organophilized) and both these clays (pristine and organophilic) were added to the matrix and to PP/sisal composites by melt compounding in an internal mixer (Rheomix 600) accessory coupled to a Haake - Büchler System 90 Torque Rheometer. Operating conditions were: roller rotors, 50 and 100 rpm for 5,7 and 10 minutes at 180 ° C. Clay and fiber contents of 3% and 20%, with respect to the matrix, were employed. Composites processed at 50 rpm for 7 min were thermally aged in an air circulating oven operating at 110 ° C for up to 15 days. Our data indicates that the composite exhibited higher moduli and lower tensile strengths than the matrix and that, as expected, better results were exhibited for the hybrids containing organoclay. Best overall tensile properties were obtained for the composites processed at 50rpm for 7 min and, under the experimental conditions adopted, there was little change in modulus and tensile strength with thermal exposure for the matrix and for the systems PP/sisal and PP/clay. Organoclay addition to these systems, however, led to heat sensitive composites whose properties significantly decreased with thermal exposure time. This behavior was associated with poor thermal stability of the organoclay.

S11-118

ON THE EFFECT OF THE CURE PROCESS ON THE MECHANICAL PROPERTIES OF STEEL FIBRE REINFORCED EPOXY COMPOSITES

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Hybrid moulds obtained by rapid tooling processes have been frequently used to reduce the time-to-market and improve product quality. The moulding blocks produced with cast resins are adopted when small batches of plastic parts are required. During injection moulding, the mould is subjected to static or dynamic loads. This means that the composite of the moulding blocks should have good mechanical performance to avoid premature failure of the mould. In spite of the poor thermal and mechanical performance of these composite materials they are being investigated for their easy processing and acceptable cost. Ferrous materials are suitable fillers for applications where improved thermal and electrical conductivities are sought. However the relatively high density with respect to the matrix causes the filler to sediment, originating an inefficient composite. In this study the mechanical properties of epoxy composites with various volume fractions of short steel fibres were investigated. The composites were poured into silicone moulds to produce tensile test specimens. The cure up to gel time was carried out using three processes: static, dynamic and in-magnetic-field. Mechanical properties were determined, and the topography of fractured surfaces analyzed. It was found that the amount of steel fibres and the cure process have significant influence on the mechanical properties. As expected, the static process leads to the sedimentation of the filler. The tensile strength decreased to almost half of the unfilled resin. The specimens cured using the dynamic and in-magnetic-field processes behave very differently: the addition of fibres raises the mechanical performance up to a maximum fibre fraction below that corresponding to the maximum volume packing ($\approx 20\%$). The in-magnetic-field process gives a performance nearly 8% superior to the dynamic process.



MECHANICAL AND THERMAL PROPERTIES OF HYDROXYAPATITE FILLED POLY(METHYL METHACRYLATE) COMPOSITES

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Poly(methyl methacrylate) (PMMA) filled with hydroxyapatite (HA) filler has been widely used in biomaterial application. Acrylic denture base material was prepared from PMMA filled with HA. This research work attempted to investigate the effects of hydroxyapatite (HA) on the tensile properties, flexural properties, fractured toughness, and thermal properties of poly(methyl methacrylate). PMMA powder was mixed with monomer of methyl methacrylate (MMA) stabilized with hydroquinone. Benzoyl peroxide (BPO) and ethylene glycol dimethacrylate (EGDMA) were used as initiator and crosslinking agent, respectively. The HA loading was ranged from 5-20%. The PMMA/HA composites were prepared by using heat-processing polymer powder and liquid method. The polymerization of PMMA/HA were carried using both water bath and heat-compression technique. The thermal properties of the PMMA/HA composites were characterized using differential scanning calorimetry (DSC), thermogravimetry analysis (TGA) and dynamic mechanical thermal analysis (DMTA). The tensile and flexural modulus of PMMA was increased by the addition of HA. This is attributed to the reinforcement effects of HA. Experimental data of tensile and flexural modulus for PMMA/HA composites was compared with several theoretical models and equations. In addition, it was found that the storage modulus (E'), glass transition temperature (T_g), and fractured toughness properties of PMMA were influenced by the HA loading.

S11-132

Investigation Gas barrier properties of PP /clay nanocomposite films with EVA as a compatibilizer prepared by melt intercalation method

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In this research work, Polypropylene (PP) nanocomposite films have been prepared by melt intercalation method and their morphology and gas barrier properties have been investigated. For preparation of nanocomposites, ethylene vinyl acetate copolymer was used as a compatibilizer for facilitating the formation of either intercalated or exfoliated nanocomposites. Morphological information was determined using X-ray Diffraction (XRD) and transmission electron microscopy (TEM). XRD spectrums and TEM images confirmed that increasing the EVA content leads to achieve intercalated nanocomposites. Also permeability measurement tests for oxygen showed that gas barrier properties of PP/EVA nanocomposite films significantly improved by introducing organoclay in the blend even in low concentration of organoclay.



Study on the structure and properties of POSS filled Polypropylene prepared by reactive blending

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Polyhedral oligomeric silsesquioxane (POSS) has been effectively incorporated into polymers by copolymerization, grafting or even blending. The octavinyl POSS grafted polypropylene (PP) was first prepared by reactive blending. The compatibility, crystalline behavior, dynamic mechanical behavior and thermal behavior of physical blending and reactive blending composites of PP/POSS were investigated. Wide-angle X-ray diffraction analysis shows that the POSS in the reactive blending composites has better compatibility with PP than in the physical blending composites. POSS in physical blending composites can act as β nucleating agent and the relative proportion of the β -form increases rapidly at lower POSS content but changes little when the POSS begins to agglomerate and crystallize (at POSS content higher than 2 wt %). The forming of β -form crystalline is mainly due to the dissolved POSS molecules but not the crystal of POSS in PP matrix. For the reactive blending composites, the β -form crystalline disappears even as the nonreactive POSS can act as effective β nucleating agent. This is probably due to the strong α nucleating effect and higher crystalline temperature. Differential scanning calorimetry analysis shows the reactive blending composites have higher crystalline temperature while POSS in the physical blending composites have little effect on the crystalline temperature. The modulus of reactive blending composites increases in the presence of POSS, while that of the physical blending composites decreases with increasing POSS content. The reactive blending composites have higher thermal stability and flame retardancy properties than that of the physical blending composites.

S11-198

Graphitic-epoxy composite bonding joints strength

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Composites are at present very well-known as constructional materials. One of their essential properties is that they can be designed according to needs. Constructional composites are the large and diverse group of compound materials. A large variety of components used when producing composites is available. Also one can find the variety of the shape and measures of the component strengthening material and different technologies of their production can be used. However all constructional composites have a common feature - large value of endurance coefficient, which can be obtained as a result of suitable components selection. Composites have a numerous applications in many industry branches. There exists the large variety in the joining methods of these materials used in different type of constructions. Very often composites are connected with other constructional materials. One of such joining methods is bonding. The technology of bonding has many advantages, among which is the possibility to connect different constructional materials having different properties and geometrical measures. The article presents research results in which graphitic-epoxy composites were used. They are among other things applied in the aircraft industry. The purpose of described researches was to examine the strength of analyzed composites bonding joints. For this purpose two kinds of graphitic-epoxy composites were used, differing only with the thickness and the kind of used fabric graphitic-epoxy to their realization. To research the strength single-lap bonding joints. Exercible of the connection one used composites samples equal to 100x20 mm and the suitable thickness depending on the kind of investigated material. The thickness of first composite was equal to 0,48 mm and in the case of second one - 0,66. The epoxy glue Loctite Fast Epoxy 3430 was used in researches. The time of the hardening carried out was equal to 48 h. As the manner of the preparation of the composites sur



Effects of process conditions on the phase structure of silica filled rubber blends

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Effects of process conditions on the phase structure of silica filled butadiene rubber(BR)/styrene-butadiene rubber(SBR) blends and silica filled vinyl butadiene rubber(VBR)/isoprene rubber(IR) blends were investigated by dynamic mechanical tests and transmission electron microscope observations. It was found that the domain size of each rubber phase in the blends decreased with decreasing the agglomerate size formed by silica particles. The agglomerate size was dependent on the sort of silica, affinity of silica with rubber and conditions for processing. The affinity of silica with SBR was better than that with BR. Further, the affinity was higher for VBR than for IR. For BR/SBR blends, a better dispersion of silica particles in the blends (smaller agglomerates) was achieved when silica was mixed with BR first, followed by the addition of SBR, resulting in the formation of smaller domain of each rubber phase. Because SBR acts as surfactant in the silica filled BR. On the other hand, for VBR/IR blends, the domain size of each rubber was not directly related to the affinity of silica with rubber. The domain size was greatly affected by the mixing torque. The higher the torque, the smaller the domain size that could be achieved. The BR/SBR blends are partially miscible, on the other hand, VBR/IR blends are completely immiscible. Such the difference might induce the effects of process conditions on the phase structure of silica filled rubber blends.

S11-239

Crystallization behavior of PBT/POSS nanocomposites

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Poly(1,4-butylene terephthalate) (PBT)/Glycidylsobutyl Polyhedral oligomeric silsesquioxane (POSS) nanocomposites have been prepared by in situ ring-opening polymerization and melt-compounding, respectively. Cyclic butylene terephthalate oligomers (CBT) was polymerized with POSS in the presence of butylchlorotin dihydroxide as an initiator to obtain pCBT/POSS composites. Commercial PBT was compounded with POSS as a control. The crystallization and melting behavior of the composites with different POSS contents were investigated by differential scanning calorimetry, and the crystallization kinetic parameters were determined according to the Avrami method. The molecular weight of the synthesized pCBT is two times higher than the commercial PBT but it decreased a lot after synthesis with the POSS. The pCBT/POSS composites exhibited similar crystallization but different melting behavior compared to those of PBT/POSS composites. The PBT/POSS composites exhibited typical multiple melting peaks which was independent of the POSS contents and crystallization temperatures. The pCBT/POSS composites exhibited multiple melting peaks only after crystallized at lower temperature. Optical polarizing microscopy analysis suggested that the presence of POSS gave rise to an increase in the number of nuclei and a decrease in pCBT or PBT spherulitic size. The average Avrami exponent n of the pCBT and composites increased with the addition of low POSS content. A significant increase in crystallization kinetic constant and a decrease in crystallization half time of pCBT were observed in the presence of POSS particles, indicating a heterogeneous nucleating effect of POSS on crystallization of pCBT. The melting temperature and equilibrium melting temperature of pCBT in the composites decreased with increasing POSS content, which is directly related to the size of pCBT crystals.



MECHANICAL BEHAVIOUR OF SILKWORM (*Bombyx mori*) COCOON

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Silk is attractive because of its strength, toughness and biological compatibility. In this research, *Bombyx mori* silkworm cocoons, which are natural silk reinforced bio-composites, were assessed compared with man-made silk reinforced polymer composites. A comparison of the structural differences and mechanical properties between natural and man-made silk reinforced composites followed.

S11-255

Rheological properties of highly filled composites of polyolefins with calcium carbonate

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The comparison of viscosity values of highly filled polyolefins composites (HFPC), with calcium carbonate as a filler, determined by off-line and on-line procedure was investigated in this work. The traditional off-line methods, like cone-plate rotational and capillary rheometers and extrusion plastometer were used. The results have been compared with up-to-date on-line method, performed directly by means of a single extruder. Three types of materials were used: blends of commercial polyolefins (HDPE/iPP); composites of HDPE/iPP/CaCO₃ (where iPP/CaCO₃ was a commercial master batch) and composites of HDPE/iPP/CaCO₃ (where iPP/CaCO₃ was a self prepared master batch, and CaCO₃ was modified by calcium stearate and waxes). The content of CaCO₃ in all the blends was in the range between 48 and 64 wt. %. A shear thinning behavior for the steady state shear flow viscosity was observed for all the samples. Moreover the viscosity significantly decreases as the CaCO₃ content was increased. This behavior was more evident in the low shear rate regime and almost disappeared in the high shear rate regime. The results have been discussed in terms of the influence of both calcium stearate and waxes on the viscosity of HFPC. Finally comparing the off-line and on-line measurements similar trends for the shear flow viscosity have been observed.



The effect of vibrations on void content in composite materials

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Defects are detrimental to mechanical properties of composite materials. Our advanced materials research group is making efforts to increase the quality of laminated sandwich composite structure by eliminating common defects such as voids, bubbles and poor adhesion at interfaces. We are experimenting and analyzing the effect of mechanical vibrations applied to the curing system of composite materials production, particularly on minimizing void content. A laboratory system has been assembled to investigate the mode of vibration, the most appropriate position for application and the ideal frequency of vibration to reduce defects to a minimum. Range of frequency of vibrations covered was from 2Hz to 8kHz, for different period of vibrations. The vibration system consisted of a shaker, power amplifier and signal generator. A flat mold was attached to the shaker's top. The composite laminates were made by hand lay-up using 12 plies of E-glass fibres and vinyl-ester resin, with thickness of around 3 mm. All laminates were made under room temperature condition, using vibrations for 10 or 30 minutes, immediately followed by curing using UV lamp, cut, polished, and examined under optical microscope (64 magnification) and SEM (Scanning Electron Microscopy) to determine types and quantity of defects. Void counting and sizing was carried out at 8 random points through the entire specimen cross-section thickness. After counting voids all data were categorized into 4 groups, depending on the size (radius) of the bubble cross-section. The results showed reduction in number of bubbles (as well as in void content) at frequencies between 10Hz and 50Hz for 30 minutes of vibrations. Longer period of vibrations indicates more efficient void deduction (grouping smaller bubbles into bigger and removing the same, and dispersing small bubbles). It is felt that the vibratory energy provided a mechanism for migration and dispersion of voids.

S11-302

Influence of PP-g-MA and OMMT contents in the thermal, mechanical and morphological properties of PP/PP-g-MA/OMMT nanocomposites

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Nowadays, the development of nanocomposites are one of the last evolutionary steps of material science technology. The isotactic polypropylene (iPP) possesses many desirable properties such as low density, high thermal stability and good resistance to solvents. The use of lamellar silicates as reinforcement filler for polymers has gained attention due to the possibility of preparation of materials with better properties with low filler content. iPP (Polibrasil HP 500N - MFI 10.1 g/10 min), PP-g-MA compatibilizer (Polybond 3200 - 1 wt% of MA) and the organically modified clay (Cloisite 20A OMMT - CEC 95 meq/100 g clay) were used to prepare the nanocomposites. The PP/PP-g-MA/OMMT nanocomposites were processed in a Coperion ZSK 26 co-rotational twin screw extruder (L/D ratio 44). The temperature profile used in the eleven zones was 180 (first to fourth), 190 (fifth and sixth), 200 (seventh to eleventh), and the screw speed was 300 rpm. The OMMT content is 0, 1, 3, 5 wt% and PP-g-MA is 0, 5, 10 wt%. The TGA analysis shows that the incorporation of OMMT in the PP/PP-g-MA matrix shift the initial temperature of degradation of PP to higher temperatures. The Young's modulus increases with the incorporation of the OMMT and decreases for higher PP-g-MA content. The PP/PP-g-MA/OMMT nanocomposite (85/10/5) showed an enhancement of 14% in relation to the PP/PP-g-MA matrix. The X-Ray diffractograms indicates that the clay d-spacing in the nanocomposites increased mainly for the nanocomposites with PP-g-MA and with 1 wt% of OMMT (3.0-3.6 Å) indicating the intercalation of the PP into the OMMT lamellas. The TEM micrographs shows that the increase in the PP-g-MA content led to a better dispersion of the clay presenting a mixture of exfoliated and intercalated morphologies.



An Innovative Method to Reduce Percolation Threshold of Carbon Black Filled Immiscible Polymer Blends

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An Innovative methodology for selective localization of carbon black (CB) at the interface of immiscible polypropylene/polystyrene (PP/PS) polymer blend in order to reduce the percolation threshold in CB filled conductive polymer composites has been investigated. In CB-PP/PS blends, CB was found to preferentially localize in the PS phase. However, upon the introduction of styrene-butadiene-styrene (SBS) tri-block copolymer, CB showed higher affinity to the polybutadiene (PBD) section of the SBS copolymer, which was selectively localized at the interface between PP and PS, within the PP phase and within the PS main particle coating PS subparticles and connecting them together. There is a 40% reduction in the percolation threshold in the (70/30) PP/PS blend upon addition of 5 vol% SBS because of the selective localization of CB in the PBD phase and the change of blend morphology. SBS was found to change the morphology of the dispersed PS phase into a rod-like structure at a critical SBS loading. Moreover, for composites with or without SBS, at a certain CB loading above the percolation threshold, the mixture exhibited a co-continuous morphology in contrast to the dispersed morphology of the (70/30) PP/PS blend without CB or at low CB loadings.

S11-353

STRUCTURAL INVESTIGATION OF GLASS/POLYSILOXAN WOVEN COMPOSITES

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In this paper has been investigated real internal structure of woven composites glass fiber/polysiloxan matrix. It is necessary to now behaviour of internal structure, what there is created during prepreg process. It is necessary to precisely identify the yarn paths in the structure and to determine the real structure. Investigated composites were reinforced with 6 layers of plain-weave fabric, their structure was studied in two mutually perpendicular cross-sections along warp or weft directions. The reinforcing yarn system can be analysed on three hierarchical levels: volume, plain and length elements. In composite volume we can observe the fabric layer sliding and mechanical interlocking of layers, distortion of fabric structure, yarn nesting, and interaction of fabric layers. Multifilament yarn, which is the basic element of woven fabric reinforcement consists of a thousands of fibers. These fiber bundles exhibit the lenticular cross – section. Microphotograph is the best way for obtaining view of internal structural imperfections. Original procedure was suggested for computer vision of woven composite, from microscope scanning to FEM modelling. Raster images are able converted to vector format. Converting is very complicated process, it needs much knowledge about computer vision and computer performance. The quantitative parameters following from the structural analysis can be utilized for the analysis and prediction of material properties. Non-destructive tests and scanning on the confocal microscope are possibilities for improvement investigation in future. This study was supported by the Czech Scientific Foundation within project No 106/03/H150 and by Ministry of Education, Youth and Physical Training VCT II – 1M4674788501.



Comparative study between simulation and experimental results of fiber orientation in LFRT.

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In this work we have done a comparative study of the long fiber orientation in LGRT under certain injection conditions and different simulated models. It was subsequently made by the statistical study with the aim of linking the guidance of process conditions (injection rate, temperature, cooling and packing time). Finally presents the results obtained through surfaces response. They described correlations between injection rate and fiber orientation, between fiber orientation and packing times and cooling time and fiber orientation.

S11-419

coupling agents in ABS/SGF composite systems

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acrylonitrile-butadiene-styrene(ABS) / short glass fiber(SGF) composites were prepared. The effect of fiber volume fraction on the properties of composite was investigated, and test results were compared with theoretical equations. SEM micrographs vividly confirmed that the adhesion between ABS and neat glass fiber is poor. Silane coupling agents were selected on the basis of their organofunctional groups to improve the interface. The role of these functional groups and the coupling agent amount was studied by comparing processability, mechanical and physical properties of the composites containing different silane-treated glass fibers. It seems that using effective coupling agents reduces the deviation of properties from theory. The affinity of different functional groups to ABS as well as the strength of chemical and physical bandings formed during the reaction of silane modification, decides the effectiveness of the relative coupling agent. The effect of coupling agent amount on the compatibility was investigated and K-values of mixture low were calculated. By properties and morphology analysis we concluded that using 3%wt Trichlorovinyle Silane results a better properties, for this composite. This conclusion is supported by the theoretical possibility of forming stronger bands at the interface, for this specific coupling agent.



Rheological, Mechanical and Tribological Properties of Nanocomposites Based on Polyamide with Functionalized SEBS Copolymers

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The rheological, mechanical, and tribological properties of nanocomposites based on polyamide 6 with various functionalized hydrogenated styrene butadiene thermoplastic elastomer (styrene-ethylene/butylene-styrene block copolymer, SEBS) were investigated. Polyamide 6 nanocomposite used as the main component of composites is a commercial product of layered silicate (Clay) filled Polyamide 6 (PA6/Clay). In this study, four types of hydrogenated styrene butadiene thermoplastic elastomer: unmodified SEBS (SEBS), maleic anhydride grafted SEBS (SEBS-g-MA), amine group grafted SEBS (SEBS-g-NH₂) and carboxyl group grafted SEBS (SEBS-g-COOH) were added with PA6/Clay composite. Polymer blends of PA6/Clay and SEBS, SEBS-g-MA, SEBS plus SEBS-g-MA, SEBS-g-NH₂ or SEBS-g-COOH (PA6/Clay/SEBS ternary systems) were extruded by a twin screw extruder and injection-molded. Dynamic viscoelastic properties in the molten state of these blends and their tensile, impact and tribological properties of these blends were evaluated. Dynamic viscoelastic properties such as storage modulus and complex viscosity were found to increase with the addition of SEBS, and were influenced by the types of functionalized groups. In particular, the containing of SEBS-g-MA has the strongest influence on these viscoelastic properties. Influence of the addition of SEBS on the mechanical properties of these systems differed for each mechanical property. Although the tensile properties decreased with SEBS, Izod impact properties were improved with the addition of various functionalized SEBS. The tribological properties such as coefficient of friction and specific wear rate were improved with the addition of SEBS, and the influence of the amount of addition was higher than the type of SEBS used. These results indicate that the new tribomaterials developed in this study have sufficient balance amongst moldability, mechanical, and tribological properties.

S11-474

Evaluation of alternative methods for the fabrication of hybrid fiber mats for polymer composites

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In this work, sisal fiber was used in partial replacement of glass fiber/unsaturated polyester composites. Hybrid composites with polyester resin and three different sisal/glass fiber compositions (25, 50 and 75% by weight) were produced. The mats were fabricated by two methods: in the first one, the mats were prepared by fluidizing by air the fibers in a chamber; in the second one, the mats were prepared by agglomerating the fibers dispersed in two different liquid media – ethanol and water. Density and water absorption were determined experimentally for pure components and the composite. Tensile and flexural tests were performed according ASTM methods. The results showed that the mats prepared with ethanol gave better homogeneity while the air process caused higher agglomeration of the fibers. The composites prepared with mats using water had shown a decrease in the glass fiber dispersion with decrease in the mechanical properties as well.



INCREMENTAL SHEET FORMING OF GLASS MAT THERMOPLASTICS

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The reinforcement of thermoplastics with glass fibers is a common practice to produce a composite material with high specific stiffness and mechanical resistance. Depending on the length of the fibers, they can be classified into continuously and discontinuously reinforced composites. The former have higher mechanical performances but the latter present a better flowability of reinforcement and matrix phases, which is a distinct advantage in processing. Most of the previous and the current research work dealing with processing glass mat thermoplastics (GMT) has focused on closed-mold technologies such as compression and injection molding. However, these processes are not suitable for small production volumes, since they require high tooling investments. Thermoforming may present a low-cost alternative but only for simple part geometries. To avoid these drawbacks, a very promising technology called Incremental Sheet Forming (ISF) has recently been introduced for sheet metal forming by using CNC machine. The major advantage of incremental forming is that it can be used to make sheet parts quickly and economically without using expensive tooling and equipments. The applicability of ISF technique to GMT sheets is presented in this paper. In particular, the study considers the effects of the main process parameters to sheet formability. To demonstrate the viability of the ISF process for GMTs some specific shapes and automotive components are used. The deformation process of GMT sheets is proved to be feasible by using a conventional equipment, such as a 3-axis CNC machine, a hemispherical tool and a simple frame. A GMT sheet is clamped in the frame and then locally heated and incrementally deformed by the rotating tool. The formability of GMTs is evaluated by measuring the thickness profile of the deformed sheets.

S11-482

Evaluation of interfacial adhesion on the mechanical properties of Polypropylene/sisal fibre composites

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Thermoplastic and thermosets matrix in composites using vegetal fibers have been receiving considerable attention as the substitute for synthetic fiber. The main reasons are that these vegetable fibers can be harvested from renewable resources, presents long aspects of their mechanical properties, low cost, low density and no health problems. However, the great problem for the success of these materials is that the vegetal fibre exhibit a high hydrophilic property as they are composed of lignocellulose, who contains strongly polarized hydroxyl groups and therefore, the fiber are incompatibles with hydrophobic polymer matrix and especially for poor and conventional resin matrices. On the other hand, there are little literature about compression molding processing thermoplastic composites, because this process is more used for thermosetting composites. The aim of this work is to use maleic anhydride grafted polypropylene (MA-g-PP) as the compatibilizer for PP/sisal fiber (SF) composites and to study the influence of temperature and composition of SF in the fiber-matrix interfacial adhesion. PP/SF and PP/MA-g-PP/SF composites were processed in a thermal compression molding machine at processing temperatures of 180, 190, 200 °C and SF composition of 20 and 30 % in weight. Composites were characterized by physical (density and water absorption) and mechanical properties (tensile test). Preliminary results of the density showed that PP/MA-g-PP/SF composites were slightly lower than PP/SF composites. On the other hand, it was observed that the increase of tensile strength of composites with the presence of MA-g-PP and it could be due to the number of polar groups introduced into matrix increased and thus improved the interfacial adhesion between the fiber surface and PP matrix. Complete results will be showed in the final work.



Effects of Interface Control of Polypropylene Composites on Thermal Stability and Flame-retardancy

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The world-wide consumption of polypropylene (PP) is the largest among polymers. However, its inflammability is a serious draw-back. In this study, in order to overcome this problem, flame-retardant poly(p-phenylene ether)(PPE) and inorganic compounds were incorporated into PP and the structure and properties of the obtained composites were investigated by XRD, SEM, TEM, DSC, TGA, and cone calorimetry. It was shown from TEM observations that inorganic compounds such as Mg(OH)₂ were preferentially located in the PPE phase, not in the PP phase in composites of PP/PPE/inorganic compounds. It was also shown that the size of the PPE dispersed phase is drastically reduced from ca. 4 microns to 0.3 microns by addition of SEBS copolymer. This is probably because the styrene block and the EB block of SEBS are miscible with the PPE phase and the PP phase, respectively, so that SEBS functioned as an excellent compatibilizer. Furthermore, organo-treatment of inorganic compounds was attempted. It was clearly shown that not only the addition of flame-retardant PPE but also the addition of a SEBS compatibilizer to stabilize the PP/PPE interface and the organo-treatment of inorganic compounds to stabilize the polymer/inorganic interface dramatically increased thermal stability. The flame-retardancy of PP evaluated by a cone calorimeter was also drastically improved by addition of PPE, SEBS, and organo-treated inorganic compounds. Thus, the present study verified that the addition of a flame-retardant polymer and interface control were very effective for improvement of both thermal stability and flame-retardancy.

S11-517

Polypropylene/pristine-clay nanocomposites by melt-compounding: compatibilization by an ionomer

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Polypropylene (PP)/clay nanocomposites are usually prepared by melt-compounding. There are synthesized using compatibilizers like PP grafted with maleic anhydride (PP-g-MA) and almost exclusively organoclay. However the organo-treatment of the clay adds a cost. Moreover, the surfactant degrades at extrusion temperatures and a collapsing of the silicate layers is reported. PP/pristine-montmorillonite (MMT) nanocomposites were here prepared by a melt-compounding process using ionomers of highly-grafted elastomeric PP-g-MA (2.5 or 3 µeq/g) and water injection. PP, PP-g-MA, sodium acetate trihydrate and pristine MMT were melt-blended in a twin-screw co-rotating high-speed extruder and water was injected directly into the melt. So, the PP-g-MA was in situ partially neutralized by the sodium acetate trihydrate during the nanocomposite synthesis. The morphology and the structure of the composites were investigated by wide-angle X-ray spectroscopy (WAXS) and transmission electron microscopy (TEM). Thermal behavior and degradation were investigated by thermo-gravimetric analysis (TGA) in air atmosphere. The pristine clay interlayer spacing was observed increased in WAXS only when the PP-g-MA was neutralized into ionomer. TEM pictures of the PP/pristine-MMT nanocomposite compatibilized with the ionomer were similar to the ones of a conventional PP/organoclay nanocomposite (made from a commercial masterbatch nanocomposite Nanoblend Concentrate MB1001 from PolyOne): a mixture of stacked and exfoliated silicate layers was observed. However, higher weight loss delay under air was measured for the PP/pristine-MMT nanocomposite compatibilized with the ionomer than for the conventional nanocomposite.



Effect of Fibre/Matrix Modification on the Mechanical Properties of Extruded Flax /Polypropylene Composite

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The flax/polypropylene compounding is characterized mechanically namely; stiffness, strength and impact in addition to the water absorption behaviour. Different approaches are experimented. The extruder screw layout is modified through the addition of one and two kneader steps. Both fibre and matrix are chemically modified. The fibre surface of one flax group was washed with sodium hydroxide solution. Others were further treated using trimethoxyvinylsilan and acrylic acid. Matrix is also modified by maleated polypropylene, trimethoxyvinylsilan and acrylic acid. The combinations of different fibre/matrix are extruder compounded, injected moulded and finally tested. At 30% flax weight content; matrix modification using MAPP and silane/MAPP show twice as strength as the untreated one, more than 300% improvement regarding stiffness and about 40% for impact. On the other hand; fibre treatment shows insignificant improvement. Water absorption also is reduced to almost the half of the untreated compound.

S11-546

VEGETAL MICROFIBRIL POLYOLEFIN COMPOSITES PREPARED BY EXTRUSION

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Composites of thermoplastics and thermosets with vegetal fibers are known since many years. Most are processed by thermoforming and show poor mechanical properties. Reinforcing agents, like fiberglass and talc, improve the mechanical properties of thermoplastics but the composites are heavier than the pure polymers. These reinforced materials are processed continuously by extrusion and, in general, the parts are molded by injection. More recently, vegetal fibers started to be investigated as reinforcing agent for thermoplastics due to their biodegradability, light weight, low abrasion and availability (renewable resources). The main aim of these works is to substitute fiberglass in these composites. Our laboratory developed a composite of polyamide-6 with a vegetal fiber extracted from the leaves of a plant named curauá (originally from the Amazon region). This composite is continuously produced by extrusion. This work is now extended to polypropylene and high density polyethylene. Composites are processed in a Coperion ZSK-26 counter rotating intermeshing twin screw extruder equipped with a side feeder and degassing. A coupling agent, polyolefin grafted with maleic anhydride, in 2 wt % concentration is used to impart the adhesion between the hydrophilic fiber and the hydrophobic matrix and, consequently, improve the mechanical properties of the composites. The equipment and screw design used proportionate not only an excellent dispersion of the fibers but also its fibrillation, which means that the fiber is split in its microfibril components. These microfibrils have an average diameter of 3 microns. This proportionate a higher aspect ratio for the reinforcing microfibril in comparison to the pristine fiber (65 to 70 microns diameter) also contributing to improve the mechanical properties of the composites. Injection molded test samples of PP and HDPE composites containing 20 wt % of fiber show mechanical properties comparable to composites made with fiberglass.



POLYMER MATRIX - FILLER INTERACTION IN THE NANOCOMPOSITES BASED ON POLYURETHANE/ POLY(2-HYDROXYETHYL METHACRYLATE) SEMI-INTERPENETRATING POLYMER NETWORKS AND ARTIFICIAL DIAMOND-GRAPHITE NANOFILLER

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Semi-interpenetrating polymer networks (semi-IPNs) based on polyurethane (PU) and poly(2-hydroxyethyl methacrylate) (PHEMA) unfilled and filled with ultra-dispersed diamond-graphite powder with particle size of 2-50 nm (produced by high shock blow method) were synthesized by sequential method. The filler was introduced into the systems during the process of PU synthesis. The thermodynamic miscibility, dynamic mechanical and physico-mechanical properties have been investigated. Dynamic mechanical analysis data of unfilled semi-IPNs have two distinct maxima of $\tan\delta$ related to polymers in their glass transition temperature domains. The results confirm that the studied semi-IPNs are two-phase systems with incomplete phase separation. The introducing of ultra-dispersed diamond-graphite filler in the semi-IPNs during process of synthesis result in essential changes in dynamic mechanical behaviour of materials especially in the temperature range of PU glass transition: the introducing just 0.25% of filler lead to decreasing of amplitude and broadening of glass transition domain of PU. For filled semi-IPNs the superposition of two glass transition temperature domains of PU and PHEMA for small content of PHEMA and full suppression of segmental motion of PU for large amount of PHEMA were observed. The mechanical properties of semi-IPN samples reflect the changes in structure with increasing amount of PHEMA in the systems. Young's modulus increases from 13 MPa up to 658 MPa with fraction of PHEMA. The introducing of ultra-dispersed diamond-graphite filler in the semi-IPN samples resulted in the significant growth in stress at break and in Young's modulus. The maximal effect have been obtained for samples with 0.25% of filler.

S11-582

Influence of processing conditions in small scale melt mixing and hot pressing on resistivity of polycarbonate -MWNT composites

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The field of polymer- CNT nanocomposites has acquired great deal of attention due to the requirements of new advanced materials with enhanced mechanical and electrical properties. Melt processing of these materials is an important and cheap way to produce large volumes of consumer and industrial products. The processing conditions can have a big influence on the nanotube network formation inside the matrix. Such a network is responsible for electrical conductivity through the composite. In this work, the influence of processing conditions in manufacturing of polycarbonate (PC) – multiwalled carbon nanotubes (MWNT) composites and pressing conditions on volume resistivity is investigated. Composites containing 1 wt% MWNT (Baytubes, Bayer MaterialScience) were produced in a small scale Daga microcompounder using different processing conditions, whereas mixing temperature, mixing time and rotor speed were varied. The extruded strands were hot pressed under comparable conditions and volume resistivity was measured. Very high differences in the resistivity values were found, although the same amount of MWNT was incorporated. Hot pressing of composite strands / granules is also very decisive. In order to characterize mechanical and electrical properties of polymer composites, generally pressed plates are used. Pre-compounded polycarbonate with 1 and 2 wt% MWNT were used to investigate the influence of pressing parameters such as pressing temperature, pressing time and pressing speed according to a three level factorial design. The volume resistivity of PC with 1 wt% MWNT compound varied by 8 orders of magnitude, whereas for 2 wt% MWNT the variation was much smaller.



Two-dimensional filling simulation of fibrous porous media

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The presence of voids in the resin transfer molding (RTM) process have deleterious effects on mechanical properties of final products. Different fluid velocities due to local permeability in the fiber bed, which result in uneven flow front, may account for void formation. In order to better understand flows during molding, a finite element scheme for simulation of fluid flow across the micro-structured fibrous media is developed in our work, which has the ability to capture the flow front and to predict incomplete filling or void formation in fiber preform of the RTM process. A volume-of-fluid (VOF) method has been implemented in Eulerian frame. Furthermore, the incorporation of the periodic boundary condition to our scheme simplifies the modeling of fiber array for the regular packing cases. We showed the flow pattern for several distinct microstructures and discussed the influence by pressure gradient, viscosity ratio and porosity which may indicate the appropriate processing conditions whereby void formation could be minimized. This work has been supported by the 2nd stage of BK21 and NURI projects.

S11-639

RHEOLOGY OF HIGHLY FILLED POLYMERIC MATERIALS

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Highly filled polymeric materials exhibit complex rheological properties. In this study the rheological characteristics of various highly filled suspension systems are evaluated. The suspension systems include hydroxyl terminated polybutadiene/calcium carbonate with bimodal particle size distribution, hydroxyl terminated polybutadiene/sugar with bimodal particle size distribution, and Exact 5361/Dechlorane. Shear dependent maximum filler volume fraction, shear thinning and thixotropic behaviors are presented.



Rheological Properties and pvT-behaviour of a Model System for PIM

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Powder injection moulding (PIM) is an important and accepted industrial technique for net shaping of precision components which may have a rather complex geometry. In order to meet the imposed, often rather strict, requirements with regard to dimensional accuracy, it is important to have an adequate knowledge and control of the rheological behaviour and the related processing properties of the powder/polymer melt (feedstock). Such a knowledge is furthermore of crucial importance in numerical simulations of the PIM-process. In the present work, a model system, consisting of steel powder, poly(ethylene oxide) and wax, is used in order to illustrate how the viscometric properties of the system can be related to the corresponding properties of the polymeric binder system. In a similar way, the pvT (pressure-volume-temperature)-behaviour of the model system is analysed and discussed. The pvT-behaviour, which has not been extensively reported on for PIM-feedstocks, is considered to be of significant relevance for controlling the outcome of the injection moulding process.

S11-735

High Strength Polymers - Transverse Crack Resistance of Fibre Reinforced Solid Phase Oriented Materials

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Combining molecular orientation with short glass fibre reinforcement it has been shown that significant improvements in material stiffness in the direction of orientation can be achieved¹. Molecular orientation within these studies was achieved by a small scale die drawing process 10 oC below the melt temperature of a composite material. The initial drawing billet being injection moulded so that preferential fibre alignment in the draw direction was achieved during the injection stage. Whilst increases in room temperature modulus and failure strain were achieved during this initial investigation it has also been proposed that the presence of short glass fibre reinforcement can significantly affect fracture toughness perpendicular to the orientation of fibres². Within this paper we will discuss the preparation of oriented filled polymer materials with fibre content up to 13 % w/w along with comparisons of fracture toughness measurements perpendicular to the orientation direction. Crack length, propagation rate and direction will also be examined and compared to theoretical models.



Preparation and Properties of HDPE/ Sugarcane Bagasse Cellulose

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The use of natural fibers as reinforcement for thermoplastics has generated much interest due to the low cost, low density, high specific properties characteristics and also due to the possibility of environmental protection when is used sugarcane bagasse residue. Another important advantage is that natural fibers are biodegradable and nonabrasive, readily available and present specific properties comparable to traditional fibers used as reinforcements into the composites, specially for automotives application for interior components as such seat frames, side panel and central consoles. In this work the mechanical and morphological properties of high-density polyethylene/ sugarcane bagasse cellulose fibers and modified sugarcane bagasse fibers composites were analyzed. Composites were produced in a thermokinetic mixer Dryser in the following composition: 5 wt%, 10 wt% and 20 wt% fibers. After mixing the samples were injection molding according to ASTM D-638 specification. Five specimens were tested in an INSTRON universal-testing machine (model-8801), pneumatic claws equipped at a crosshead speed of 10 mm.min⁻¹. Specimens were tested in tensile mode and composite fractures surface were analyzed in an LEO 1450 V scanning electron microscopy with tungsten filament operating at 20 kV, utilizing low vacuum technique and secondary electron detector. Results showed that high-density polyethylene/ modified sugarcane bagasse fibers composite presents better mechanical performance than that reinforced with sugarcane bagasse cellulose. However it was observed a poor interaction between fiber and matrix due to the formation the fibers agglomeration, which induced specimen ruptures of the specimen. These effects can be observed by the morphological characteristic of materials.

S11-873

Effects of Interfacial Compatibilizer upon the Microstructure of PLA/clay Nanocomposites: Permeability/Biodegradability Relationship

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Interfacially compatibilized polylactide (PLA)/clay nanocomposites have been fabricated via melt mixing process. Effects of the maleic anhydride grafted polypropylene (g-PP) as interfacial compatibilizer upon the developed microstructure and the dispersion state of the clay nanolayers have been evaluated by performing X-ray diffraction spectrometry (XRD) and transmission electron microscopy (TEM). It has been found that the g-PP structural parameters such as melt viscosity and graft degree influence the degree of intercalation and exfoliation of the clay nanolayers. Effects of the feeding routes (direct and masterbatch) of the materials during the mixing process upon the composite microstructure have also been investigated. Dynamic melt rheological characterization showed nonterminal as well as nonlinear viscoelastic behaviour within the low frequency range, indicating the formation of a network structure by the dispersed clay nanolayers throughout the PLA matrix. The Nanocomposite samples with lower oxygen permeability exhibited enhanced biodegradability, which can be attributed to the higher interaction sites between the nanolayers and the PLA matrix and therefore more catalytic hydrolysis of the PLA chains. This indicates that dispersion state of the clay nanolayers plays an important role in the rate of biodegradation of the PLA matrix. The prepared PLA nanocomposites showed much higher potentiality to be used for the production of biodegradable packaging films with high oxygen impermeability compared with the pristine PLA samples.



WATER ABSORPTION AND TENSILE PROPERTIES OF KENAF BAST FIBER- PLASTICIZED POLY(LACTIC ACID) BIOCOSMOSITES

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Increasing awareness on the environmental safety coupled with new rules and regulations has forced manufacturers to consider biodegradable materials for their products. The aim of this work was to investigate tensile properties and water absorption behavior of biocomposites from kenaf bast fiber and poly(lactic acid). The composites were prepared by Haake internal mixer and compression molding. Composites containing fiber loading up to 40 wt% were prepared. PLA is brittle and would result in composites with poor impact properties. Because of this, a blend of PLA and a plasticizer, polyethylene glycol (PEG) at 10 wt% was evaluated as the polymer matrix and referred to as p-PLA. p-PLA/KBF composites exhibited good tensile properties than unfilled p-PLA. The tensile strength and modulus of the composites increased with increasing fiber content due to high fiber-matrix compatibility. Reinforcing effect of KBF was observed when fiber loading exceeded 20 wt%. Scanning electron microscopy (SEM) of the tensile fractured surfaces revealed a very good adhesion between KBF and p-PLA matrix. In addition to fiber pullout, failure by fiber fibrillation was also observed. Water absorption behavior of p-PLA/KBF composites were studied by immersion in distilled water at room temperature for 60 days. All composites as well as unfilled p-PLA were found to exhibit non-Fickian behavior. The deviation from Fickian water uptake behavior was attributed to the development of micro-cracks on the surface and inside the materials. In addition to micro-cracks that occurred along the fiber length, water absorption too caused fiber-matrix debonding. Both were revealed via SEM examination on the surfaces of the composites. p-PLA became very brittle after water absorption. SEM examination too showed micro-cracks on the surface of p-PLA. Results suggested that KBF is a potential reinforcement for plasticized poly(lactic acid) composites but possible applications should avoid high humidity environment.

S11-976

Biocomposites Materials Based on Lignocellulosic Fibres and Thermoplastic Matrices

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The processing of polymeric materials reinforced with renewable raw materials, such as natural fibres, has received a considerable attention during last years. This study deals with the preparation of lignocellulosic composites by reactive extrusion processing in which good interfacial adhesion is generated by a combination of fibre modification and matrix modification methods. Despite several advantages of using micropowder from cellulosic fibres as reinforcement in biocomposites there are some limitations. The most important drawback is the poor compatibility between the hydrophilic polysaccharides and the mostly hydrophobic polymer matrix. Hence, the biofibre powder - matrix interface is usually the weakest point in a biocomposite which makes the performance of the final composite limited. The best results were obtained with flax surface PVOH grafting. The feature was confirmed by the appearance of transcrystallinity in isothermal crystallisation experiments run in a hot stage of a polarized optical microscope. The effect of silane and PVOH solution processed lignocellulosic materials i.e. flax and wood micropowder, as well the addition of maleic anhydride modified polyethylene compatibilizer on the thermal stability of the composites was investigated. The DSC scans were taken in the temperature range from 50 °C to 350 °C with the programmed heating rates 40°/min, 20°/min, 10°/min. The heating curves at 10°/min merges into the exothermic crystallisation peak resulting some stable crystals present of the liquid phase formed. Esterification of Ma-PE-cellulosic fibres-PE analysed by FTIR-spectroscopy. The peak at 1746 cm⁻¹ arise from ester bonds between the copolymers and fibres, whereas the peak at 1739 cm⁻¹ from monomeric form of dicarboxylic acids. The composite materials were manufactured with mixing in twin screw extruder having fibre content 10, 20 and 30 wt%. The results indicated that mechanical properties are strongly dependent from fibre loading.



Impact Modified Polystyrene Based Nanocomposites: Production by Melt Intercalation and Characterization

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Polystyrene (PS) and three different organically modified clays (Cloisite® 15A, 25A and 30B) were used to produce nanocomposites by melt intercalation. SEBS-g-MA was used to function as impact modifier and compatibilizer between PS and the organoclays. The ternary nanocomposites were prepared using a twin screw extruder, by diluting with pure PS in the second extrusion run, to obtain the desired compositions. In addition to ternary composites, binary materials (PS/Organoclay and PS/Elastomer) were also prepared to observe the effects of clay and elastomer alone. After processing the nanocomposites, they were characterized by XRD, SEM, DSC and mechanical tests. X-Ray diffraction analysis showed approximately 40 % increase in the basal spacing of the organoclay in PS/Cloisite 25A binary composites. However, in ternary composites containing Cloisite 25A, exfoliated structure was observed for both 1 and 2 wt % clay loading. This implies that the elastomer plays an efficient compatibilizer role by interacting with Cloisite 25A and PS matrix. According to the tensile test results, the highest improvement was obtained for the ternary nanocomposite containing 2 wt % Cloisite 25A and 5 % SEBS-g-MA, having the highest tensile strength and elongation at break values among the compositions studied. Moreover, Young's Modulus values of the ternary nanocomposites increased in all clay types.

S11-1030

Fiber Breakage in Carbon-Fiber-Reinforced Nylon 6/Clay Nanocomposites

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Short carbon-fiber-reinforced nylon 6/clay nanocomposites were prepared via melt compounding, and fiber breakage and dispersion during processing have been investigated. It has been found that the presence of organoclay can improve fiber dispersion, which is due to dispersion at the nanoscale of exfoliated clay sheets with large aspect ratio. The bimodal distribution of fiber length is observed in fiber-reinforced nanocomposites, which is similar to that in conventional fiber-reinforced composites. The improvement of fiber breakage at moderate organoclay loadings is also observed, which is ascribed to the rheological and lubricating effects induced by organoclay.



NOVEL BIOCOMPOSITES. USE OF SEAWEED RESIDUES AS FILLER IN POLIOLEFINS.

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Morphological and chemical structure of seaweeds residues (SR) allowed inferring their high potential as biomaterial, due to their interconnected porous structure that might induce the osteoconductive process. In this way, this work proposes the development of composites that combine the SR properties with polymeric matrices, such as polyolefins, specifically polyethylene (PE) and polypropylene (PP). Composites were elaborated in an internal mixer varying the processing conditions, respect the polyolefin used in order to obtain better dispersion. Different SR concentrations (0-30 ppc) were added to the matrix at the selected mixing conditions. The characterization was carried out by electron microscopy, melt flow index, differential scanning calorimetry, termogravimetric analysis and tensile properties. The best SW-PP dispersion was obtained at 80 rpm, 180 C and 3 min, while in the PE the optimal conditions were 90 rpm, 160 C and 5 min. In both cases higher mixing rates and lower temperatures made favorable the homogeneous dispersion, which was corroborated through the micrographs by the diminishing of agglomerates. Regarding to the filler incorporation both polymer displayed an increase of the viscosity as SR content raised, however properties such as tensile strength and elongation at break decreased as result of the poor interaction between the SR particles and matrices. For this reason, a coupling agent, i.e NZ12, was added in other to improve the interfacial interaction, which only was observed in PE composites by the increase in the Young Modulus. Therefore, it was necessary to include other coupling agent (acrylic acid) on PP composites, finding a synergistic effect which allowed improving the mechanical properties. Concerning the thermal characterization, in all cases, it was found that neither SR nor coupling agent inclusion, did not affect the crystallization and melting temperatures. However it was noted a slight increase in the thermal stability.

S11-1102

Injection moulded polycarbonate composites with chemically functionalized carbon fibres

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Carbon fibres (CF) have been widely used as reinforcement of polymers for the improvement of their mechanical and electrical properties. The main problem with CF reinforcement is their low surface energy and poor interaction with polymer matrix. In the present work, CF are functionalized using chemical methods that were successfully applied to carbon nanofibres [1]. The chemical functionalization is expected to improve the CF dispersion and its interface with the polymer matrix, enhancing the composite properties. The composites obtained are studied for their mechanical and electrical properties. The dispersion and interaction of the CF/polymer matrix is studied using scanning electron microscopy. [1]. R. Araújo, M. C. Paiva, M. F. Proença, C. J. R. Silva, ""Functionalization of Carbon Nanofibers by 1,3 - Dipolar Cycloaddition Reactions and its Effect on Composite Properties"", Composite Science and Technology, 2007, 67, 806-810.



The effect of carbon nanofibres on the properties of carbon fibre/polycarbonate composites

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Carbon fibre (CF) polymer composites have been widely used in structural applications. The main issue in short fibre composites is the chemical inertia of CF surface that difficult fibre dispersion and lead to poor fibre/matrix interaction. Similar problem arises in the formation of composites with carbon nanofibres (CNF). CNF present excellent mechanical and electrical properties, at a higher cost compared with CF. In the present work CF and CNF were functionalized using the same chemical method [1]. Polycarbonate composites with 10% CF, functionalized and non-functionalized, and 10% CF with 1% CNF, both functionalized, were prepared by injection moulding. The influence of functionalization on the CF/CNF/PC interface is analysed by scanning electron microscopy. The improvement in mechanical and electrical properties resulting from the addition of a small amount of CNF is presented. [1]. R. Araújo, M. C. Paiva, M. F. Proença, C. J. R. Silva, "Functionalization of Carbon Nanofibers by 1,3 - Dipolar Cycloaddition Reactions and its Effect on Composite Properties", *Composite Science and Technology*, 2007, 67, 806-810.

S11-1131

Mechanics of Random-Fiber Networks: A Direct Simulation

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The interactions between fibers in a random-fiber network transmit stress, cause fiber curvature, and influence fiber orientation in the process of many types of composites. A few theories describe the mechanics of fiber networks, but almost no simulation results are available. Here we report a direct numerical simulation of the mechanical behavior of random-fiber networks. The finite element method is used, and each fiber is represented by a small number of 3-D beam elements. The calculations assume a periodic structure, to avoid boundary effects, but within the unit cell the fibers are placed randomly. A special algorithm creates an initial structure of straight, random, non-intersection fibers, from which a unit cell with periodic boundary conditions is built automatically. The simulation uses an explicit time integration of dynamic equations, with a general contact algorithm (ABAQUS/Explicit). A typical run involves 700 fibers with $L/D = 100$, compressing the network from an initial volume fraction of 5% to a final volume fraction of 25% using 10^5 time steps. At the final volume fraction there are 11,000 fiber-fiber contacts. Results from the simulation are in rough agreement with the Van Wyck (1946) theory for compaction pressure. They show good agreement with Toll's (1999) theory for number of fiber-fiber contacts, and they also show good agreement with a simple slender-body model for fiber orientation, at least during initial uniaxial compression. This simulation provides an interesting tool for understanding the mechanics of random-fiber networks, and building models of composite materials processing.



Characterization of carbon nanotube (CNT) dispersion in thermoplastic materials

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The use of carbon nanotubes as filler in polymer materials is success promising regarding the optimization of mechanical, electrical, flame resistance and barrier properties. The properties of such composites depend in a high manner on dispersion and distribution of CNT in the matrix. The most economical way for the production of such composites is compounding by melt mixing. The key factors for achieving an optimal degree of dispersion and distribution of CNT are the parameters of the compounding process. In previous works the method of the online measured electrical conductance was presented as a tool for the characterization of the dispersion process and kinetics during the compounding process of carbon black filled rubber mixtures. Recently, we have adapted this method to the melt mixing process of CNT filled polycarbonate (PC). Because of the special viscoelastic properties of the PC melt, an optimization of the conductance sensor configuration in the internal mixer was necessary. With the optimized equipment, we have investigated the influence of technological parameter, i.e. chamber temperature, rotor speed, fill factor, and material factors, i.e. CNT content, on the CNT dispersion and distribution in dependence on the mixing time. Samples were taken out of the process at different mixing times, and the morphology was analyzed by optical microscopy and atomic force microscopy. The correlation between the kinetics of the mixing process, the CNT dispersion and the properties of the compounds will be discussed.

S11-1203

Characterization of Poly (vinyl alcohol) Copolymers and Wood Dust Composites, termed as Green Composites

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The wood industry and agribusiness produce large amounts of biomass. This biomass is one of the most abundant sources of natural polymer (cellulose) and its utilization not only add value to one of the most versatile raw materials (wood), but also contributes to reduce environmental concerns regarding the disposal of waste wood. Wood dust can be added to plastics, ceramics, paints and molds to enhance their surface properties [1]. In our previous work we developed composites based on wood dust and PVA. Composite films were cross linked as well wood dust was modified to enhance the mechanical and thermal properties [2]. In the present work we have used the copolymer of PVA (Poly (vinyl alcohol-co-ethylene) containing 27% ethylene. The as-synthesized composite materials are typically characterized Fourier-Transformation Infrared (FTIR) spectroscopy and, wide-angle x-ray diffraction. The mechanical analysis of the composite material was studied by tensile test. The morphological image of as-synthesized materials was studied by optical microscope (OM). REFERENCES:[1].Yamanaka T, Yano H, Watanabe T, Honda Y, Kuwahara M. Bulletin of the wood research institute, Kyoto: Kyoto University, 2000, 25[2] Bana Ruchi, Banthia A.K, Polymer-Plastics Technology and Engineering, Volume 46, Issue 9, September 2007 , pages 821 - 829



Effect of sawdust surface treatment and compatibilizer addition on mechanical and morphological properties of polypropylene/sawdust composites

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Polypropylene/sawdust composites were investigated to assess the effect of sawdust surface treatment and compatibilizer addition on polymer/fiber adhesion. Two silane coupling agents were used for sawdust surface treatment (vinyl-tris(2-methoxy ethoxy)silane and 3-amino propyl triethoxy silane). Maleic anhydride grafted polypropylene was used as compatibilizer. Composites were prepared in a co-rotating twin screw extruder coupled to a Haake torque rheometer and submitted to tensile and bending tests, differential scanning calorimetry (DSC) as well as scanning electron microscopy (SEM). Results showed that incorporation of untreated sawdust to PP caused reduction in composite tensile strength and increase in stiffness. When the only treatment used was surface modification with silane coupling agents, no significant changes were observed in mechanical properties. However, when compatibilizer was added to the composites, tensile strength was increased and % elongation at break reduced, indicating improved system compatibility. The composite presenting the highest increase in tensile strength was that containing sawdust treated with amino silane in addition to the compatibilizer. SEM analyses corroborated the mechanical property results. Sawdust and compatibilizer addition caused reduction in crystallinity degree of polypropylene.

S11-1275

Polypropylene composites reinforced with coconut fibers

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The use of the composite polymeric materials have been increasing mainly in the manufacturing of automotive components. The amazing chemical and physical properties of these materials prompts the development of a new class of materials. Although the better mechanical and chemical properties, these materials have a environmental problems related to the discard. To solve these problem a new research and development were carried out through the natural based fibers like coconuts and sisal fibers as well as sugar cane pulp. These fibers embedded in a polymeric matrix prompts properties values very close to the synthetic related fibers. The natural fibers are environmentally friendly, been easy to recycle and biodegradable. These study deals with recycled polypropylene composites, reinforced with coconut fibers using as coupling agent based on maleic anidrid (MAPP) graphitized polypropylene. The fiber addition effects on the mechanical and thermal properties were tested in recycled and pure polypropylene.



Development and characterization of thermal and mechanical properties of nanocomposites based on recycled polypropylene reinforced with hybrid short sisal fiber/nanomineral filler

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The polymeric nanocomposites materials can be seen as an possibility enhance the polymeric properties, to supply different industrial segments. A new material class based on hybrid organic and inorganic materials, has been developed through the polymeric matrix nanocomposites like polypropylene and natural sisal as mineral nanofiller. Development this work was carried out through the nanocomposites hybrid sisal fiber/mineral nanofiller polymeric matrix with world wide industrial application as engineering materials. These material are based on a natural reinforcement that can be renovated like sisal fiber and put together with a mineral filler as nanometric particles. The synergy of these components can results in a specific properties, enviromental friendly because they are ease recycle and can easy biodegradate. The hybrid nanocomposites products was compared with mineral nanofiller-recycled polypropylene, and short sisal fiber recycled polypropylene, through to heat thermal distortion (HDT) tests, soften vicat point tests, and tension strenght, as well as, impact energy absorption.

S11-1290

Resin Film System Modified by Nano-elastomer for Resin Film Infusion (RFI) Process

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A novel high-performance resin film system modified by nano-elastomer, suitable for resin film infusion (RFI) process, has been developed. DGEBA liquid epoxy was modified by nano-elastomer and isocyanate resin with 4,4'-diaminodiphenyl sulfone (DDS) as the curing agent. In order to improve the thermal properties of the film, different concentration of bismaleimide (BMI) were added. Differential Scanning Calorimeter (DSC), Rheometer and Thermogravimetric analysis (TGA) were used to examine the effects of nano-elastomer and BMI added amount on the glass transition temperature and thermal properties. The influences of nano-elastomer concentration on the interlaminar shear strength (ILSS), Mode I and Mode II fracture toughness of cured composites were discussed as well.



Particulate polymer composite materials with controlled mechanical properties

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In order to improve the final physical and mechanical properties of composite materials a porosity control of the material microstructure is strongly required. In this paper the necessity of continuous improvement of the final microstructural properties of composite materials is investigated through a multiple approach which concerns not only the appropriate composite mix design and the optimum particle size distribution, but also the better moulding. In particular, a control of the basic technological factors of the final materials microstructure can be reached through control of vacuum, mechanical pressure and vibration during shaping. This study represents the first step toward a technological development in the field of the industrial implementation process.