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Şirketimiz, bir taraftan yurt dışında üretim yapan firmaların Türkiye temsilciliği görevini üstlenirken, diğer taraftan **AR&GE** çalışmalarını yürütmekte ve **moleküler biyoloji, moleküler genetik** ve **hücre kültürü** alanlarında hizmet vermektedir.

AR&GE DEPARTMANIMIZ

Şirketimiz, 2003 yılında ODTÜ-TEKMER' de araştırma-geliştirme faaliyetlerine başlamıştır. 2005 yılı itibari ile T.C. Sağlık Bakanlığı izni ile üretime başlayan şubemiz, 2007 yılında laboratuvarını daha da genişleterek **HACETTEPE-TEKNOKENT'** e yerleşmiştir.

Özellikle ülkemize know-how sağlayacak ve dünyada rekabet gücü kazandıracak olan tek nükleotid mutasyon analiz sistemlerinin (SNP) geliştirilmesi çalışmaları 2006 yılında başarıyla tamamlanmıştır. Bu yıldan itibaren 5' Nükleaz Yöntemine dayalı **Tip I Mutasyon Analiz Sistemleri** ve Agaroz Jel Elektroforezine dayalı **Tip II Mutasyon Analiz Sistemleri** geliştirilerek, CE sertifikalandırılması yapılmıştır. Son olarak 2009 yılında ürünlerimize **Tür Tayin Analiz Sistemi** eklenerek, günümüzün en önemli sorunlarından olan işlenmiş et ürünleri içeriğinin PCR ile tespiti sağlanmıştır. Şu anda FMF için multipleks Tip I Mutasyon Analiz sistemi için çalışmalar devam etmektedir.

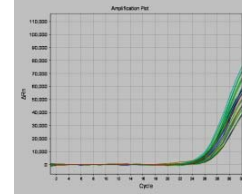
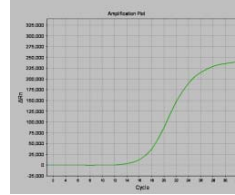
TEMSİLCİLİKLERİMİZ

Temsilciliğini yürütmekte olduğumuz firmaların "üretici" olmalarına ayrı bir önem verilmektedir. Bu firmalardan ithal edilen rutin ürünler, özellikle biyolojik ürünler (enzim gibi), öncelikle kendi laboratuvarımızda lot bazında test edilmekte ve test sonrası araştırmacıların kullanımına sunulmaktadır. Temsilciliğini yapmakta olduğumuz firmalar;

Amresco Inc. ♦ **Biological Industries** ♦ **Serva GmbH** ♦ **Corning Costar** ♦ **Bioron GmbH** ♦ **Eurofins MWG Operon** ♦ **Syngene** ♦ **Cleaver Scientific Ltd.** ♦ **Boeco** ♦ **Bioteke Corporation**

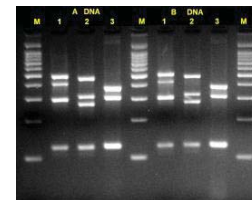
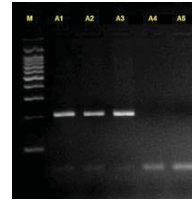
ÜRÜNLERİMİZ

Tip I Mutasyon Analiz Sistemleri :



- ♦ **FII Prothrombin**
- ♦ **FV Leiden**
- ♦ **FV Cambridge**
- ♦ **FVR2 (1299)**
- ♦ **MTHFR 677**
- ♦ **MTHFR 1298**
- ♦ **TNF 1031 T/C**
- ♦ **FMF**
- ♦ **JAK2 V617F**
- ♦ **PAI 4G/5G**
- ♦ **Hemokromatozis**
- ♦ **Kistik Fibrosis**
- ♦ **Tür Tayin**

Tip II Analiz Sistemleri:



- ♦ **Y mikrodelsiyon 8 ve 15 bölge**
- ♦ **Y mikrodelsiyon 8 bölge Multipleks**
- ♦ **ACE I/ D**
- ♦ **Tür Tayin**

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The Preparation and Mechanical Behavior of Polyethylene-Clay Nanocomposites

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Abstract- This study investigates the hardness and tensile strength characteristics of polyethylene (PE)/nano-clay composite. It is found that the weight percentage (wt %) of nano-clay greatly affects the mechanical properties of the composite because of the intercalated and flocculated structure of the nano-clay in the composite for higher clay loading. The hardness and tensile strength increase when the percentage of nano-clay is increased in the composite.

The dispersion of nano-clay particles into the polymer matrix can usually result in three general types of composite materials. The first type is separated polymer/clay composite, in which the polymer and clay particles remain immiscible. The second type is the intercalated polymer/clay composite, which is formed when one or more molecular chains of polymer take space in the interlayer. The third type is the exfoliated or delaminated polymer/clay composite that are formed when individual clay nano-particles are fully dispersed in the polymer matrix. Among these three structures, the exfoliated polymer/nano-clay composite show the best mechanical properties because they possess large aspect ratios, homogeneous dispersion of clay and huge interfacial areas between the polymer and clay [1, 2].

Polymer/nano-clay composite have shown excellent mechanical properties compared to the matrix polymers, including tensile strength, yield strength, elastic modulus, toughness and fatigue [3–5]. However, an understanding of the mechanical properties, such as hardness and tensile strength properties of polyethylene (PE)-clay nanocomposites is lacking. It is therefore meaningful to investigate their mechanical properties looking at the effects of the addition of different amounts of organo-clay.

PE/clay nano-composite systems were prepared by using a single screw extruder. Firstly, the clay sample was modified by adsorption of Cethyl trimethyl ammonium bromide (CTAB) based on cation exchange capacity (CEC) of clay. Modification of clay surface is monitored with zeta potential and contact angle measurements. After determining the optimum CEC value for organo-clay, the PE-clay composites were prepared at different clay contents. The samples were characterized by structural examinations, X-ray diffraction analysis (XRD), hardness measurements and tensile tests.

The XRD and tensile strength results are presented in Figure 1 and Figure 2, respectively. As can be seen from Figure 1, the main peak for organo-clay disappears after polymer intercalation. This significantly affects the mechanical behaviour of the composite (Figure 2).

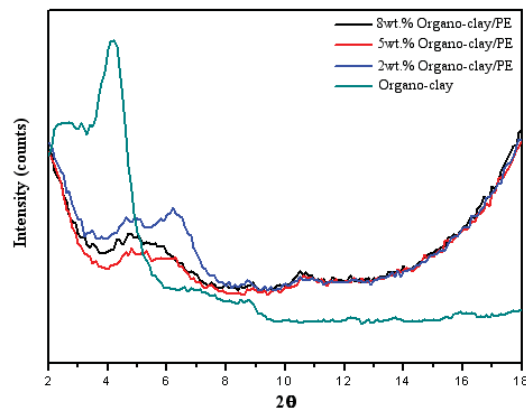


Figure 1. XRD patterns of organo-clay and nano-composites at different clay contents.

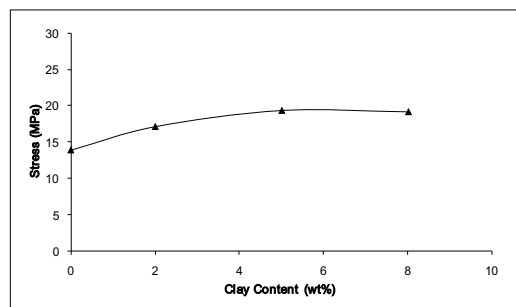


Figure 2. The variation of tensile strength of PE based nano-composites with respect to their clay content.

The present investigation reveals that there are considerable effects of the addition of organo-clay on the mechanical behavior of the composites.

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Ultrasound Sonication of Tokat Reşadiye Montmorillonite and its Feasibility in Nano Clay Production

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Abstract-We used montmorillonite obtained from Samsaş, Tokat Reşadiye where one of the biggest national clay mineral reserves exists. Particle size reduction was achieved by using a two-level ultrasound sonication, varying in acoustic power, amplitude and period, and by altering the duration of application. Our results are quite promising for the new industrial uses of nano sized Tokat Reşadiye montmorillonite.

Smectites are an important class of clay minerals because of their specific properties such as high cation-exchange capacity, adsorption, high surface area, and swelling behavior. The most widely used smectite is montmorillonite (MMT), in which a sheet of octahedral alumina (O) is sandwiched between two sheets of tetrahedral silica (T) to form TOT platelets. The technological application of clay minerals demands their improvement by decreasing in particle size [1, 2]. Particle-size reduction has been traditionally obtained by grinding (either wet or dry) [3]. Grinding invokes substantial changes of the specific surface area, structural changes, partial or complete amorphization of the powdered materials [4]. Very recently, the use of sonication has been receiving attention, as alternative to grinding, for particle-size reduction of clay minerals [3-6].

In the experimental study, Tokat Reşadiye (TR) clay minerals were processed in a laboratory type ultrasonic processor. The first group of samples, TR108, TR116, TR124 and TR148, were produced by treating the raw TR-MMT with relatively lower ultrasonic energy (acoustic power: 105 Wcm⁻²; amplitude: 125 µm; period: 1.0), but second group of samples, TR208, TR212, TR216 and TR224, were first sieved below 32 µm and then ultrasonicated at higher energy (acoustic power: 460 Wcm⁻²; amplitude: 210 µm; period: 0.8). The durations of sonication were applied as 8, 16, 24, and 48 hours for the first group of samples, and 8, 12, 16, and 24 hours for the second group of samples, respectively. The results of ultrasonicated samples, one raw TR-MMT, one TR-MMT sieved below 32 µm and one commercial Na-MMT, named as Nanocor® (NC) from AMCOL Int. Company, were evaluated and compared for their suitability for further industrial applications.

When compared to that of NC obtained as unimodal in a much narrower range, the particle size distribution of raw TR-MMT was found bimodal in a range from 0.2 µm to 101.5 µm where 90% of the particles was less than 50.8 µm. For the first group of sonicated samples, the results showed that 24-hour sonication is the threshold period of time to shift the particle size distribution to a lower range under the mild process conditions so that the distribution about the first local maxima became more intense and the frequency about the second mode decreased significantly (Fig. 1). The preparation of sieved TR clays below 32 µm was quite successful to develop a cost effective ultrasonication process, also providing a great saving in time, in order to meet particular requirements. Thus, particle size distributions of the second group of samples were achieved to include considerably high percentages of both small micron and submicron (nano) particles. Particle size percentages in nano scale (≤1000nm) were obtained as 67.2% for TR208, 63.8% for TR212, 51.1% for TR216. TR224 comprises completely nano sized particles in a narrow range (97.5%).

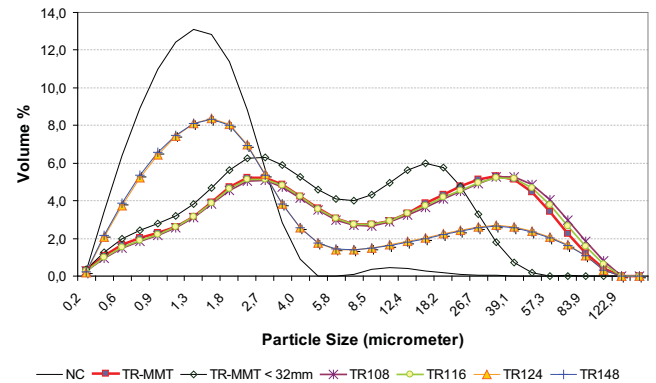


Figure 1. Particle size distributions of the samples obtained from Malvern Mastersizer-X2000 [Particle size percentages in nano scale (≤1000nm); 30.0% for NC, 7.5% for TR-MMT, 9.0% for TR-MMT<32µm, 6.8% for TR108, 6.9% for TR116, 17.7% for TR124, 18.2% for TR148]

The effect of ultrasonication is based on the significant delamination and lateral size reduction of clay minerals. The results of Brunauer–Emmet–Teller (BET) and pore analyses indicate compatible high surface values of TR clays and the significant increase in adsorption pore volume by the ultrasonication. XRD results also showed that the sonication of TR-MMT decreased the intensity of the *d*(001) diffraction indicating particle size reduction and exfoliation.

Table 2. Results of surface area and pore analyses

Sample	BET/Langmuir surface area (m ² g ⁻¹)	NLDFT cum. pore vol. (cm ³ g ⁻¹)	BJH cum. adsorption pore vol. (cm ³ g ⁻¹)
NC	30.3/94.3	0.055	0.060
TR-MMT	35.4/111.9	0.063	0.075
TR124	19.7/49.0	0.038	0.081
TR224	37.0/110.1	0.059	0.092

In summary, we showed that TR-MMT mineral is quite compatible to obtain nanoclays for new application areas. This work is based on the research project MAG107M126 funded by The Scientific & Technical Research Council of Turkey (TUBITAK). We thank Istanbul Kultur University for financial support.

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Synthesis and Characterization of Polyaniline and Polyaniline-Clay Nanocomposites

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Abstract— In this work, polyaniline (PAni) and polyaniline/clay nanocomposites have been prepared in the presence of ammonium peroxy disulphate as initiator by the method of in-situ chemical oxidative polymerization. Organically modified montmorillonite clay was obtained from Edirne/Enez region of Turkey and added to the polymer matrix at various ratios. Finally, spectral, electrical, morphologic and thermal characterizations of the nanocomposites were performed. Results show that, however clay containing nanocomposites have lower electrical conductivity, they have exhibit improved thermal properties comparing with the pure polyaniline.

Electronically conductive polymers have emerged as a new class of materials called “Synthetic Metals”. They combine the advantages of ease in processing with the good chemical and mechanical properties, while showing electronic properties of both the metallic and the semiconducting materials. They can conduct the electricity in their own matrix [1]. These amazing properties have made possible to use conducting polymers in stealth or electrostatic charge dissipation, Electromagnetic Interference Shielding, etc. technologies [2].

Among the conductive polymers, Polyaniline is the most attracting conducting polymer due to its environmental stability, processing and controllable conductivity since they have reversible doping and dedoping ability [3].

Composite materials are obtained by the combination of two or more materials. Nanocomposites are composite materials which are formed by dispersing the nano-sized particles (at least one dimension), in polymer matrix [4]. Layered silicates are the most interesting layered host because of their high “aspect ratio”. The most common particle type used in the polymer nanocomposites is the “Montmorillonite” (MMT), which is the major content of the Bentonite [5].

There are three types of nanocomposites depending on the nature of components and the method of preparation. Microcomposites are the traditional composite structure in which polymer chains cannot intercalate into the clay layers. They are also called phase separated structures. Nanocomposites are obtained in two different structures; intercalated and exfoliated. Intercalated structure in which a single (and sometimes more than one) extended polymer chain is intercalated between the silicate layers resulting in a well ordered multilayer morphology built up with alternating polymeric and inorganic layers. When the silicate layers are completely and uniformly dispersed in a continuous polymer matrix, an exfoliated or delaminated structure is obtained. XRD can be used to identify intercalated structures. In such nanocomposites, the intercalation of the polymer chains usually increases the interlayer spacing, in comparison with the spacing of the organoclay used, leading to a shift of the diffraction peak towards lower angle values [6].

In this work, we synthesize PAni-MMT nanocomposites by in-situ chemical oxidative polymerization. The organically modified montmorillonite has been obtained from Edirne/Enez region of Turkey. The characterization of PAni and PAni-MMT composites was performed by using FTIR, DT/TGA, XRD, SEM techniques. In addition, electrical conductivity was measured by four probe technique.

From the results of FTIR analysis, we found out that polymerization yielded formation of polyaniline and

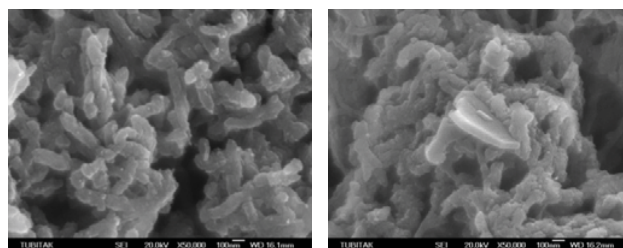
Polyaniline-MMT nanocomposite instead of Polyaniline-MMT blend.

The thermal properties of the samples were analyzed by DT/TGA instrumentation. The degradation of nanocomposites was completed in three stages. The results show that MMT nano-sheets can improve the thermal stability of the polymer matrix since they behave as a barrier against thermal degradation.

The structural characterization of the nanocomposite was done by using XRD spectrum. We found that the clay peak shifted to the lower values as the d spacing values were increasing. These results show that the structure of the polyaniline MMT clay nanocomposites are intercalated nanocomposites.

Finally we investigated the electrical conductivity of the nanocomposites. The decrease in electrical conductivity is a result of the insulating property of the clay layers which are dispersed in the polymer matrix.

The SEM photographs are given in the Figure1. From the photos we can see the interaction between the PAni and clay clearly.



Polyaniline

Polyaniline-MMT

Figure1. SEM photographs of Pure PAni and PAni-MMT nanocomposite.

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Effects of Compatibilizer Type and Processing Parameters on Mechanical Properties of Polypropylene-Clay Nanocomposites Prepared by Melt Mixing

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Abstract-The effects of compatibilizer type and the processing conditions on polypropylene-clay nanocomposite preparation by melt extrusion were investigated. The objective of the study was to optimize the nanocomposite formulation and manufacturing parameters to obtain enhanced physical and mechanical properties. The clay content, compatibilizer type screw speed and screw design of the extruder, loading order of the compatibilizers were those parameters. Considering the mechanical test results, the effects of the processing parameters on the physical and mechanical properties of the nanocomposites were discussed.

Nanocomposites are a new class of materials showing enhanced physical, thermal and mechanical properties compared to neat polymer and conventional mineral filled composites due to the much stronger interfacial forces between the nano-sized domains. Polymer-clay nanocomposites became widely studied with Toyota's work on the exfoliation of clay in nylon-6 in the early 1990s [1,2]. The main principle in manufacturing polymer-clay nanocomposites is to separate the stacked layer structure of clay aggregates and then separate individual silicate layers in the polymer. In that way, the number of reinforcing components increases dramatically since each clay particle contains hundreds or thousands of layers and the engineering properties of these individual clay layers will function more effectively. As a result, properties of the neat polymer will be significantly improved even with a very low filler loading [3]. From the nanostructural point of view, two types of polymer-clay nanocomposites are possible: Intercalated and exfoliated nanocomposites. The former is obtained when polymer is regularly inserted into the clay layer galleries and increases the gallery spacing. Exfoliated nanocomposites are obtained when the clay layers are individually dispersed in the polymer matrix [4-6].

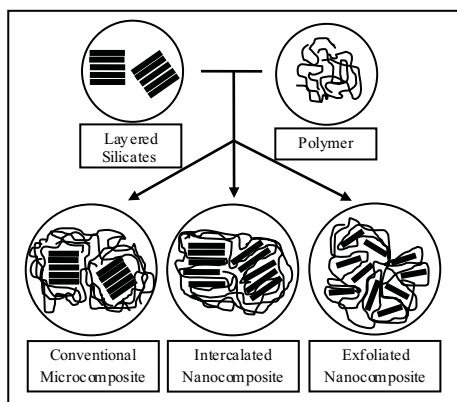


Figure 1. Types of polymer-clay composites according to the interaction between layered silicates and polymer.

This study was an experimental approach to polymeric nanocomposite world where the effects of compatibilizer type and the processing conditions on polypropylene-clay nanocomposite preparation by compounding within the twin screw extruder were investigated. The objective of the study was to optimize the nanocomposite formulation and

manufacturing parameters to obtain enhanced mechanical properties.

The optimum clay (organo-modified montmorillonite (OMMT)) content and the compatibilizer type were determined after a series of experiments. Screw speed and screw design were varied in order to obtain different shear rate profiles in the extruder. Loading sequence of the ingredients was also a parameter that affects dispersion of the clay within the polymer matrix. The compounds prepared by melt extrusion were injected in a mold to form test specimens. The specimens were mechanically tested.

Considering the test results, maleic anhydride grafted Polypropylene (PP-g-MA), as the compatibilizer, had an effect to increase strength and maleic anhydride grafted styrene ethylene butadiene styrene (SEBS-g-MA), as the compatibilizer, was better at improving ductility of the final nanocomposite. Using them simultaneously in the compound with an optimum content optimized the mechanical improvement due to their synergistic effects. Different shear profiles were tried by varying screw speed and screw design of the twin screw extruder. Processing with lower screw speed and with the screw profile which could introduce medium shear force instead of high shear, provided comparatively better mechanical results. There was only little enhancement in mechanical properties by varying the loading sequence of the different type of compatibilizers into the extruder.

In summary, according to the results of the experiments, the compatibilizer and rubber toughener has a significant effect on the level of reinforcement achieved. The process conditions for nanocomposite preparation are effective only when the good interaction between the surface of the nanoparticles and matrix is achieved. This work was supported by TUBITAK under project number 106T073. The authors also thank Research and Development Center in ARCELİK.

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Rheology of Nanoparticles in Polymeric Liquids; Tribology and Composite Applications

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Abstract-Polymeric nanocomposites containing carbon and silica based nanoparticles were produced to investigate resulting relation between the rheological and tribological properties. Evaluation of rheological properties indicate non-newtonian flow behavior; namely, shear thinning and shear thickening depending on the colloidal interactions between filler particles and liquid molecules. Shear thinning polymeric nanocomposites were found to act as a lubricant therefore it reduce friction coefficient due to its rheological characteristics.

Several type of nanoparticles; each with its own size, shape, structure and resulting properties are widely used as an additive in many applications. Recently nano particle containing lubricants are of great scientific interest since the tribological properties of lubricant become increasingly size dependent. Many researchers have tried to improve the tribological characteristics of lubricants made of nanoparticles and base lubricant to decrease friction coefficients and wear rates. Also there has been significant effort to understand the mechanism of the nanolubrication and different explanations have been given; mending effect, rolling effect, ball bearing effect, colloidal effect, protective film, and third body material transfer are some of them.^[1] Fan et al.^[2] indicate that the hydrodynamic interaction between two colloids mediated by non-adsorbing polymer chains is very important in terms of the lubrication and friction. Their numerical results show that surface-to-surface separation distance is minimum as the particles get closer therefore the effective viscosity decreases and finally approaches the lubrication regime, where the friction equals that of two close-approached spheres in a pure solvent. Therefore it is obvious that the most important parameter for the rheological behavior of nanolubricants is the colloidal interactions between filler particles and polymeric fluids.

This study systematically investigates the effects of physicochemical parameters on the rheology of nanoparticles integrated polymeric fluids to shed a light on the fundamental understanding of the rheological-tribological relationships for the nano-lubricants which is an ongoing controversial issue in the relevant literature. Firstly, we studied the effect of constituent parameters such as particle size, concentration which has already produced promising results in literature. In addition to this, surface chemistry of particles and polarity of polymeric phase was investigated. Because colloidal interactions between filler particles and polymeric liquid determine the surface to surface separation distance which is important in terms of the viscosity and friction.

To be able to study the effect of the colloidal interactions on the rheological behaviour; hydrophobic/hydrophilic fumed silica, carbon nanotube and graphene in the continuous liquid phase with different degrees of polarity have also been studied with using rotational rheometer. It was found that colloidal interactions between particles and continuous phase can be tailored by modifying the surface chemistry of particles or changing the polarity of the continuous phase. The relative strength of the interactions between particle-liquid and particle-particle determine whether dispersion is shear thickening or shear thinning.

Interaction between particles cause trapped of the fluid within floc structures. Most of the network bonds are disrupted and relatively smaller size isolated flocs form when shear applied on system. Also Figure 1a indicates that trapped

liquid molecules release, viscosity of the system decrease and flow shows thinning behavior. When the shear is ceased, small flocs interact to form a space filling network reversibly. Figure 1b shows polymer chains have strong affinity for the particle surface consequently some of polymer chains (white ones) adsorbed on the surface of particle aggregates and clusters in system at stationary condition while others (red ones) are free between colloidal particles. Most of the shear thickening fluid show slight decrease in viscosity before thickening region in shear profile. Alignment of non-adsorbed polymer chain along the flow direction facilitate flow of dense aggregates. In shear thickening regime, these aggregates and clusters are broken down and dispersed on the application of high shear fields therefore effective volume fraction of particle increase in the system due to finely dispersed small particles with adsorbed polymer chain. The polymer adsorption is essentially reversible, polymer molecules are desorbed from silica particles after shear is ceased. Dynamic light scattering were performed to measure the hydrodynamic radius of nanoparticles that are average 250 nm in shear thickening fluids and in shear thinning fluids 800nm also micron size particles were measured. These results were supported by transmission electron microscopy.

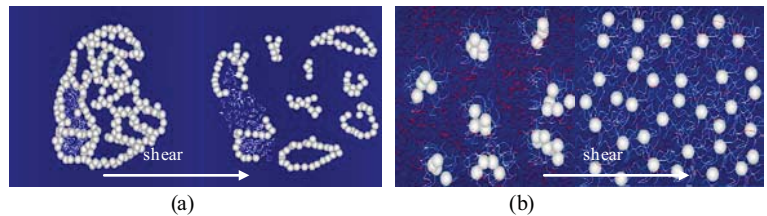


Figure 1. Schematic representation of (a) shear thinning (b) shear thickening

An ability to predict tribological properties with the help of the rheological characteristics of lubricants for a given filler and liquid matrix type is desired. These results shows that interparticle attraction between particles and particle-liquid molecules determine whether shear thickening or shear thinning flow behaviour observed. This conclusion is important because shear thinning fluids has power to reduce friction and wear whereas shear thickening fluids not.

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EPDM/clay nanocomposites: the effects of blending conditions on the properties

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Abstract- In this study, layered clay/EPDM nanocomposites were prepared by using Na⁺ montmorillonite (MMT), EPDM and MA-g-EPDM (maleic anhydride grafted EPDM). The effects of the surface modification of the clay particulates and the blending conditions on the thermal, physical and mechanical properties of the nanocomposites will be presented within the paper.

Polymer-layered silicate nanocomposites have generated significant research interest due to enhanced properties including tensile strength, solvent resistance, flame retardancy, thermal stability and permeability [1-5]. Ethylene propylene diene terpolymer (EPDM) is an unsaturated polyolefin rubber and has become extensively used in industry such as automotive tire sidewalls, cover stripes, wires, cables, hoses, belting, footwear, roofing barriers and sporting goods [3]. However, it is incompatible with polar organophilic clay to prepare products having desired properties because EPDM does not include polar groups in its backbone [2] and homogenous dispersion of the silicate layers in EPDM is difficult to realize [4]. The homogeneous dispersion of the organophobic layered clay mineral in a polymer matrix is possible through approaches that promote favorable interactions between the matrix and the silicate surfaces [5].

MMT was modified by octadecylamine to obtain hydrophobic surfaces, diminishing the surface energy, making the silicate layers compatible with polymer and ease the dispersion of inorganic clay in polymer matrix. EPDM/clay nanocomposites were prepared by a direct melt compounding method by blending of 5-10 wt. % of the clay particulates within the polymer matrix using rheometer which was followed by the addition of ZnO, stearic acid, sulphur and vulcanization accelerators by using a roll mill. The effects of the blending time, temperature and rotor speed on the properties were investigated.

The microstructural investigation revealed that dispersion of OMMT within the EPDM matrix is affected by the nanocomposite blending conditions. The mechanical test results showed that the properties of nanocomposites are significantly improved with the addition of OMMT (Fig.1). The effects of the processing conditions were manifested in both the morphology and mechanical properties, which showed significant increase when optimized process conditions are applied.

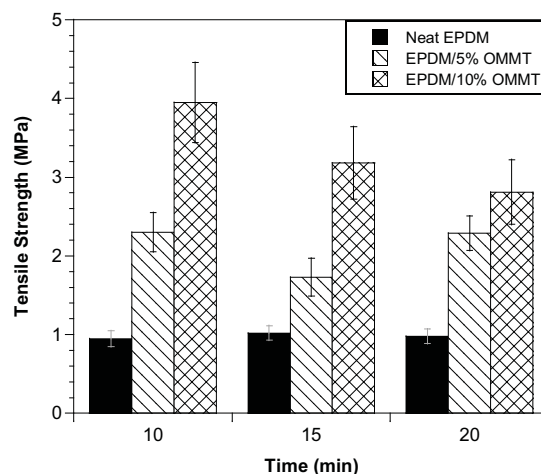


Fig. 1. Tensile strength values of neat EPDM and its nanocomposites containing 5 and 10 wt. % of OMMT with respect to various blending times.

This study was supported by TÜBİTAK.

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Synthesis and properties of Polyimide-silver nanocomposite containing Pyridyl moieties in the main chain

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Abstract-A new thermal stable polyimide-silver nanocomposite containing pyridyl moiety in the main chain was synthesized by a convenient ultraviolet irradiation technique. A precursor such as AgNO₃ was used as the source of the silver particles. Polyimide 3 as a source of polymer was synthesized by polycondensation reaction of 2,5-diamino pyridine 1 with pyromellitic anhydride 2 in the presence of iso-quinoline solution.

The resulting composite film was characterized by FTIR spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetry (TGA) and differential scanning calorimetry (DSC).

With the development of nanotechnology, the investigation on polyimide-based nanocomposites or hybrids with inorganic particles such as metals, metal oxides, zeolites, organoclay and carbon nanotubes have been explored to further enhance the properties of PI materials [1-5]. Also incorporating monomer containing heterocyclic pyridine ring into the polymer backbone is one of the most efficient methods of improving the thermal stability of polymers [4-5]. In this article a new polyimide (PI)-silver nanocomposite containing pyridyl moiety in the main chain was prepared by using ultraviolet irradiation technique at room temperature. Polyimide 3 was synthesized by reaction of an equimolar mixture of diamine 1 with pyromellitic dianhydride 2 in *m*-cresol solution and in the presence of *iso*-quinoline as a base (Figure 1).

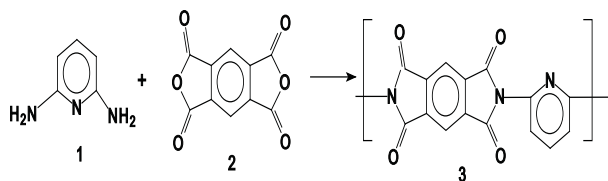


Figure 1. Synthetic route of PI 3

The FT-IR spectrum of PI 3 exhibits characteristic absorption peaks for C=O unsymmetrical stretching of imide groups (at 1774 cm⁻¹), C=O symmetrical stretching of imide groups (at 1709 cm⁻¹) and C-N stretching of imide groups (at 13840 cm⁻¹). These bands show PI 3 has been successfully synthesized. In addition, no obvious difference between the infrared spectra of the pure PI 3 and the PI-silver nanocomposite 3a was observed. The XRD pattern of the soluble PI-silver shows a broadened peak at about 30 is corresponding to the phase of PI. The four diffraction peaks in the XRD patterns of samples 3a widen greatly, indicating the formation of the nanometer scale of silver particles in the PI-silver nanocomposite. The SEM micrograph of the PI-silver nanocomposite 3a in Figure 2 shows that the silver nanoparticles were homogeneously dispersed in polyimide matrix. The thermal properties of pure polyimide 3 and polyimide-silver nanocomposite 3a were investigated by TGA and DSC experiments. The temperature of 5 and 10% weight loss and also the char yield at 600 °C of polyimide-silver nanocomposite 3a were higher than the pure PI 3. The higher thermal stability of nanocomposite 3a can be attributed to the presence of inorganic silver nanoparticles into the polyimide matrix.

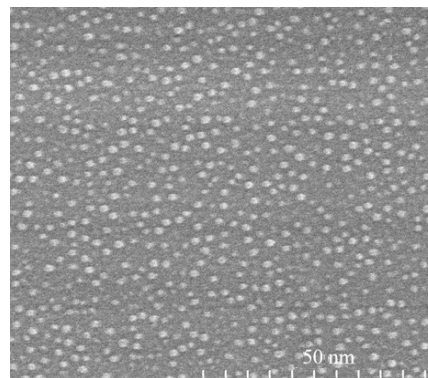


Figure 2. SEM image of polyimide-silver nanocomposite 3a

In this work, a polyimide-silver nanocomposite film was successfully prepared by a convenient reduction of silver by ultraviolet irradiation technique. The high char yield of this nanocomposite film can be related the presence of inorganic silver nanoparticles in the polyimide matrix. These properties can make this nanocomposite attractive for practical applications such as processable high-performance engineering plastics.

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Synthesis, Conductivity and Dielectric Characterization of Salicylic Acid-Fe₃O₄ Nanocomposite

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Abstract- We report on the synthesis of water dispersible salicylic acid capped Fe₃O₄ nanocomposites via a co-precipitation route. The capping of salicylic acid around Fe₃O₄ nanoparticles was confirmed by FTIR spectroscopy.

In this work we present on the synthesis of water soluble salicylic acid-Fe₃O₄ magnetic nanocomposite. To the best of our knowledge this is the first report on its preparation and magnetic and dielectric characterisation.

We successfully synthesized water dispersible salicylic acid-Fe₃O₄ nanocomposites, where the attachment of the salicylate entities to nanoparticle surface was found to be via the bridging interaction of carboxylate oxygens in Fig. 1 through FTIR analysis.

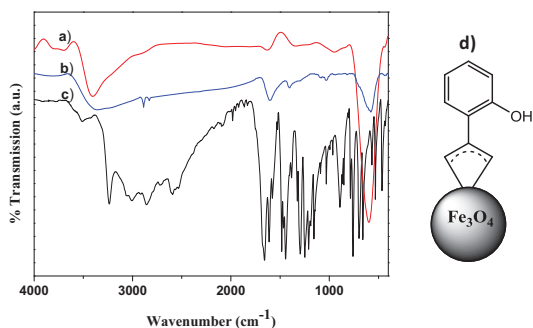


Figure 1. FTIR spectra of (a) Fe₃O₄ NPs (b) Salicylic acid coated - Fe₃O₄ nanocomposite (c) Salicylic acid and (d) Suggested linkage of salicylic acid to iron oxide surface.

XRD analysis confirmed the synthesized material as magnetite and an average crystallite size of 13±6 nm was calculated by x-ray line profile fitting. As compared to the particle size of 20 nm obtained from TEM analysis (Fig.2) these particles show polycrystalline nature.

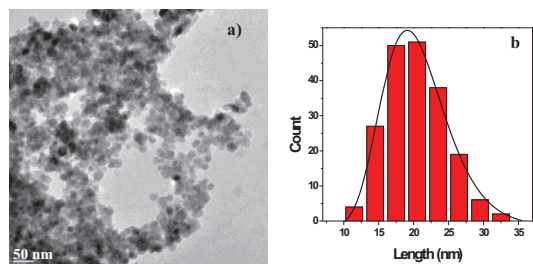


Figure 2. (a) TEM micrograph of salicylic acid coated-Fe₃O₄ nanocomposite, and (b) calculated histogram from several TEM images with log-normal fitting.

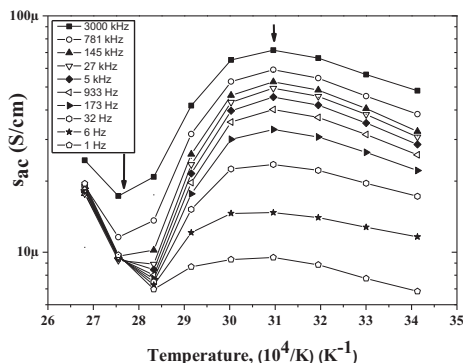


Figure 3. AC conductivity versus frequency dependence for salicylic acid-Fe₃O₄ nanocomposite from RT to 100°C.

Ac and dc conductivity measurements (Fig. 3,4) revealed semiconductor conduction characteristics [1], and various trends were observed, as a function of frequency and temperature, revealing different mechanisms dominating based on the temperature dependant reorganization of the nanocomposite.

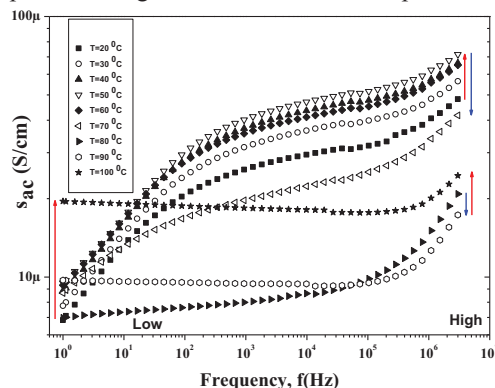


Figure 4. AC conductivity of salicylic acid- Fe₃O₄ nanocomposite versus reciprocal temperature within the frequency range of 1Hz to 3MHz.

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Functionally Filled Polymer Nanocomposites: A Novel Class of Flame-Retardant Materials

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Abstract- This study presents some of the successful attempts to improve the reaction to fire of thermoplastic polymer materials with the use of the nanocomposites approach. The potential synergistic action between filler nanopartcles and flame-retardant functional fillers were exploited to attain satisfactory reductions in the rates of heat release during simulated fire scenarios on bench-scale fire tests. Improvements in the fire properties were usually accompanied by superior mechanical properties with the incorporation of filler nanoparticles at very low loading levels in polymers.

Polymer nanocomposites attract great research interest owing to the modification of polymer properties in an exceptional manner with the use of very low filler loadings. In particular, the high flammability of polymeric materials can be significantly reduced via the “nanocomposites” approach together with the incorporation of conventional flame-retardant additives in polymer matrices. Synergistic improvements in polymer flame retardancy were reported when appropriate combinations of filler nanoparticles such as nanoclays and conventional flame-retardant additives like phosphorus or mineral-based compounds are used [1].

In this study, we present some of our attempts which have been successful in enhancing the properties of functionally-filled (flame-retarded) polymers via the “nanocomposites” approach. Organically modified layered-silicates (nanoclays) were dispersed at the nano-scale in flame-retarded polyamide-6 (FR-PA6) and high-impact polystyrene (FR-PS) resins by shear mixing via twin screw extrusion and high-power sonication-assisted solution blending. Nanomorphological characterizations were done by wide-angle X-ray diffraction and transmission electron microscopy. Exfoliated and intercalated clay nanomorphologies were obtained in FR-PA6 [2] and FR-PS [3], respectively.

Reactions to fire of the developed materials were assessed by bench-scale cone calorimeter analysis whereas flammabilities were determined by limiting oxygen index (LOI) measurements. Formation of nanocomposites enabled lower rates of heat release (Figure 1), especially the rates of peak heat release (PHRR), from the materials during combustion in a simulated mild to intermediate fire scenario (Table 1). The reductions in PHRR reached almost 40% with the use of very low levels of filler nanoparticles. Nanocomposites required higher oxygen concentrations (LOI) to support flaming combustion indicative of lower ignitability and flammability. Improvements in flame retardancy were accompanied by remarkable stiffening as inferred from the reported elastic moduli (E).

Table 1. Flame retardancy and mechanical properties

	PHRR (kW/m ²)	LOI (% O ₂)	E (GPa)
FR-PA6	255	24.9	2.5
Nanocomposite	190	30.9	3.1
FR-PS	325	19.3	2.1
Nanocomposite	200	21.1	2.9

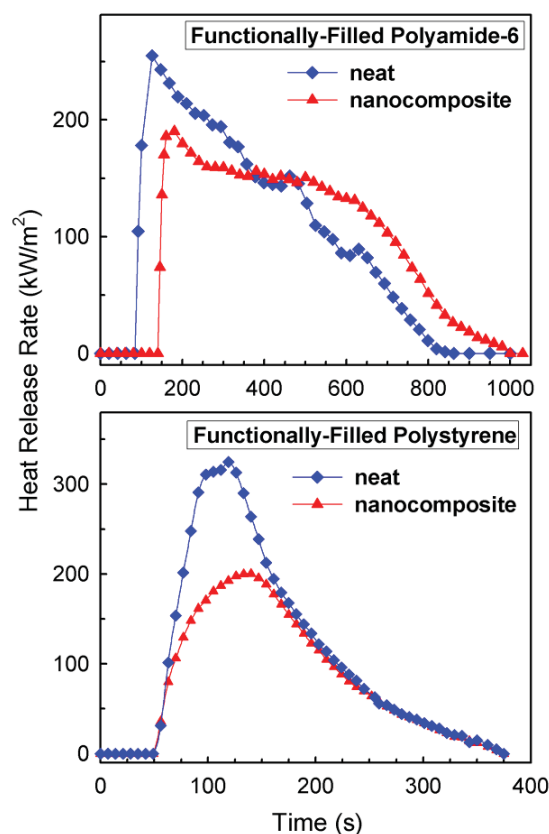


Figure 1. Reaction to fire of developed materials.

This study was supported by TUBITAK, The Scientific and Technological Research Council of Turkey; under Grant No. 107M347.

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Measurement of Interatomic Forces between Colloidal Particles and Comparison with Relevant Theories Mehmet Polat

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Interactive forces between colloidal particles lie at the heart of the adsorption, flotation, stability, rheology, plasticity and related phenomena. The Atomic Force Microscopy-Colloid Probe (AFM-CP) Method is used to obtain real-time and in-situ interaction force data in colloidal systems. However, measurement of these forces and their analysis is a tedious task with numerous details and possible pitfalls. Also, comparison of measured forces with relevant theory could be carried out using numerical analysis since an analytical solution of the Poisson-Boltzmann Equation was possible only for weakly charged surfaces which exclude most natural systems. In this paper, we present a detailed analysis of the AFM-CP measurements using α -alumina surfaces in aqueous solutions as an example. Also, we introduce a new analytical solution of the PB Equation for arbitrarily charged surfaces and show how it can be employed for analyzing the measured force data.

DLVO theory states that the interactions between the particles of a colloidal dispersion are determined by electrical double layer and van der Waals forces [1,2]. The van der Waals interaction pressure for two interacting plates is given as:

$$F_{vdW}(h) = \frac{A_{12}}{6\pi h^3} \dots\dots\dots 1$$

The electrostatic interactions between colloidal particles owe their presence to the potential gradient $\psi(x)$ between the surfaces which arises spontaneously due to charging of the particles in solution (Figure 1). The relationship which gives how the potential profile $\psi(x)$ changes in solution as a function of distance x from the solids's surface is called Poisson-Boltzmann equation:

$$\frac{d^2\psi(x)}{dx^2} = \frac{\kappa^2 RT}{zF} \sinh\left[\frac{zF\psi(x)}{RT}\right] \dots\dots\dots 2$$

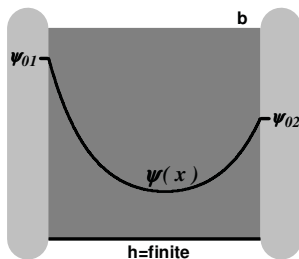


Figure 1. Potential profiles developed between two surfaces.

Unfortunately, solution of Equation 2 is possible only for weakly charged systems. Numerical analysis is used for arbitrarily charged systems encountered in most natural systems. However, recently, a new analytical technique which allows calculation of both the potential profile and the electrostatic force for such surfaces was developed [3].

The Atomic Force Microscope (AFM) can be employed to measure actual forces between colloidal particles (Figure 2). In a force measurement, the cantilever is brought close to a point on the surface progressively and a plot of cantilever deflection versus vertical piezo translation is obtained (Figure 2). This information can then be transformed into a plot of interaction force versus surface separation by proper algorithms. If the tip of the cantilever is replaced by a colloidal particle, the measured force would be that acting between the colloidal particle and the surface. This method is called the Colloid Probe Method after Ducker et al. [4,5].

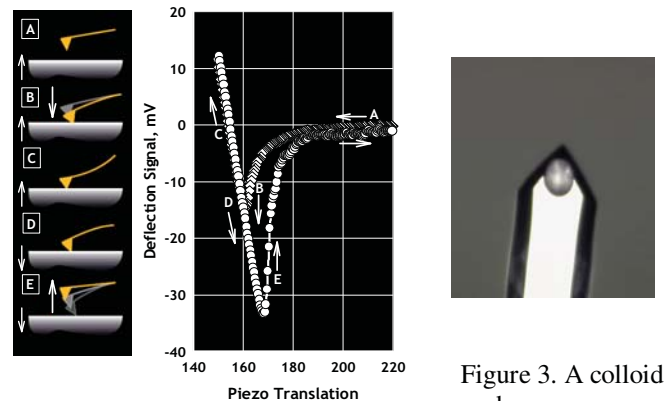


Figure 2. The force curve from AFM.

Figure 3. A colloid probe.

Force measurements were carried out between two α -alumina surfaces; a flat sapphire surface and a spherical α -alumina particle (colloid probe) [6]. Selected data from these measurements (at pH 3.5) was utilized to illustrate the use of colloid probe method in this paper. The colloid probe used is given in Figure 3. The force-distance curves obtained were compared with the DLVO theory with the help of the new analytical solutions of the Poisson-Boltzmann Equation. The results show excellent agreement as given in Figure 4.

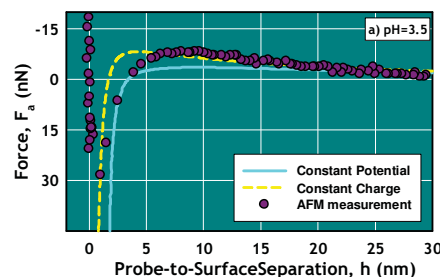


Figure 4. The measured and theoretical interaction forces between α -alumina probe and sapphire surface.

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Biodegradable Polymer- Silica Nano Composite Surface Coating for Protection of the Marble

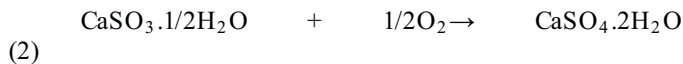
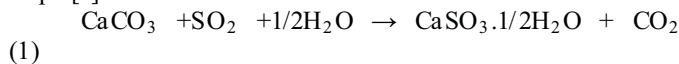
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Abstract-In this study, the protection abilities of the nanocomposite coatings were investigated. Marble slabs were coated with pure polylactic acid (PLA) biodegradable polymer and PLA/fumed silica bio-nano composite surface coating agents at different concentrations. Due to the significant effect of hydrophobicity on the coatings, contact angle analyses were defined on the uncoated and coated marble surfaces. Water capillary absorption and water vapor permeability tests, color alterations of uncoated and coated marbles were performed. Later coated and uncoated marble slabs were placed in the reaction chamber which included nearly 8 ppm SO₂ gas with 100% relative humidity to determine the protection performance under acidic conditions. Sulphation products were determined by using ion chromatography (IC). After the SO₂-Calcite reaction the morphological change of coated and uncoated marbles surfaces were investigated scanning electron microscope (SEM). As a result the protection abilities of the PLA were enhanced with the addition of fumed silica.

In the last century, increase of the industrialization was induced to many environmental problems like air pollution. Especially, sulphur dioxide (SO₂) which is the one of the most significant deterioration agent for marble monuments has been accelerated [1]. In addition that another important deterioration agent for the marble monuments is the water action [2]. SO₂ and calcite (CaCO₃) only reacted with the presence of the water and this reaction take place in the two steps [1].



In the first step of reaction, sulphur dioxide (SO₂) reacts with marble which includes calcite crystals (CaCO₃) and converts it into calcium sulphite hemi-hydrate (CaSO₃·1/2H₂O). Calcium sulphite hemi-hydrate is known as an unstable product and it easily oxidized to gypsum (CaSO₄·2H₂O) by the oxygen in the presence of water. In the previous studies, the coating agents provided some protection but they caused other problems such as removal from surface [1]. Therefore, the studies continued either looking for more environmental friendly surface coating agent or modifying coating materials with the addition of nanomaterials such as clay and fumed silica. They determined the polymer/nanomaterials nanocomposites as more effective than neat polymers [2, 3] in the surface protection.

Biodegradable polymers showed promising new materials on the protection studies. In addition they fulfill the principles accepted by International Conservation Community of Historic Monuments and Buildings. PLA is one of biodegradable polymer which showed good water and gas barrier properties and have potential to enhance the protection properties of the coatings [1].

In the first part of the study, we defined the water action effects for PLA fumed silica nanocomposites. For this purpose, capillary absorption and water vapor permeability tests were studied for uncoated, pure PLA coated and PLA/fumed silica nanocomposite coated marbles. Uncoated and PLA and PLA/fumed silica coated marbles were compared with each other by capillary water absorption and water vapor permeability tests.

Hydrophobicity takes place a significant role to determine water effects on the coating materials and we measured the hydrophobicity abilities of the coated and uncoated marble

slabs by using contact angle test. These analyses indicated that nano particles addition increased the hydrophobicity of the composites. Contact angle measurement results were presented in the Figure 1 for uncoated, pure PLA biopolymer coated and PLA/fumed silica nanocomposite coated (at 2%, 5% and 7% concentrations) marble slabs. Addition of the nano particles increased the hydrophobicity of the PLA biopolymers up to 5% silica nano particle addition.

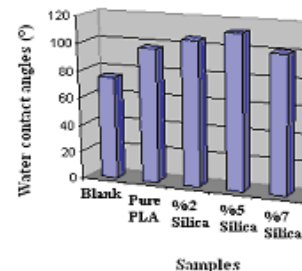


Figure 1. Contact angle test results for coated and uncoated marbles

Calorimetric measurements were used to determine color alterations of the coated and uncoated marbles. Color variation of the coated and uncoated marbles just only related with the esthetic perspective. The addition of nano silica in polymer was not caused any significant color alterations between the coated and uncoated marbles. All coatings were determined as clinically acceptable.

In the second part of the study, coated and uncoated marble slabs were taken place in the reaction chamber which included nearly 8 ppm. SO₂ and 100% R.H. Deterioration product which was the gypsum crust thickness for the coated and uncoated marble slabs were determined. Sulphate amounts which were formed on the marble surface for coated and uncoated marbles were determined from the IC results. Also, surface morphologies and formation of the sulphation products were determined with SEM.

It can be concluded that the addition of nano silica enhanced the protection properties of the biodegradable polymers when we assessed the results which were obtained from the all parts of the study. Nanocomposites were defined as excellent to protect the marble monuments.

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Preparation and properties of Polyurethane-organoclay nanocomposites Using Modified Montmorillonite with Ionic Liquids

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Abstract—In this study, segmented polyurethane (PU)/clay nanocomposite has been synthesized by polyurethane and organoclay. 1-methyl-3-octyl-imidazolium tetrafluoroborate (IL-3) was used as swelling agents to treat Na⁺-montmorillonite and for forming organoclay through ion exchange. The nanometer-scale silicate layers of organoclay were completely exfoliated in PU in the cases of 1, 3, 5 and 10% IL-3-mont/PU nanocomposites as confirmed by differential scanning calorimetry and transmission electron microscopy studies. The segmented structures of PU were not interfered by the presence of the silicate layers in these nanocomposites as evidenced by their glass transition measurements.

Polymer composites were widely used in electronic and information products, consumer commodities and the construction industry. In these polymer composites, inorganic materials were used to reinforce polymers with the idea of taking advantage of the high heat durability and the high mechanical strength of inorganic and the ease of processing polymers. Clays have been extensively used in the polymers industry either as reinforcing agent to improve the physicochemical properties of the final polymer or as a filler to reduce the amount of polymer used in the shaped structures, i.e., to act as a diluent for the polymer, thereby lowering the economic high cost of the polymer systems [1].

The polyurethanes are an important and very versatile class of polymer materials. With desirable properties, such as its high abrasion resistance, tear strength, excellent shock absorption, flexibility and elasticity. However, there also exist some disadvantages, for example, thermal stability and barrier properties. To overcome the disadvantages, research on novel polyurethane / Na⁺-montmorillonite nanocomposites is being developed in our group [2].

In a majority of these studies, clay particles were exfoliated by soft segment polyols prior to reaction with isocyanates [3]. Polyurethane chains carrying –NCO end groups were allowed to react with –CH₂CH₂OH groups of imidazolium ions in clay. Both high viscosity and clay–polymer reactions were found necessary for exfoliation of clay particles.

In this study, new polyurethane–clay hybrid materials were prepared with polyurethane based 4,4-diisocyanate diphenylmethane and ethylene glycol, and using modified montmorillonite with 1-methyl-3-octyl-imidazolium tetrafluoroborate.

The molecular structure and morphology of the prepared polyurethane–clay hybrid nanocomposites were characterized by FTIR, SEM, TGA, DSC and x-ray. The thermal and physical properties of the prepared hybrid nanocomposites were studied and correlated with the molecular structure.

Polyurethane was mixed with 50 ml of toluene solvent, and stirred at room temperature for 1 h. The mixture of PU and solvent was blended with a known amount of organoclay and stirred for 3 h at room temperature. The mixture was waited for 1 week at room temperature. After drying continued at 80 °C for 8 h.

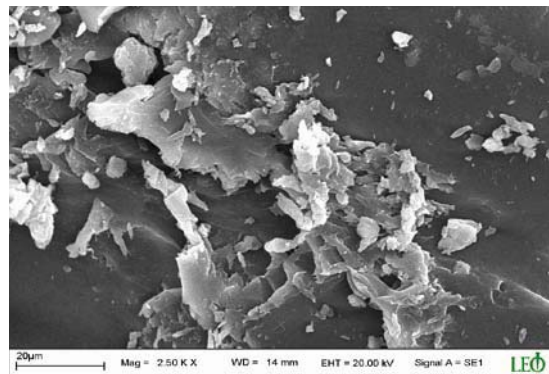


Figure 1. SEM image of PU–organoclay nanocomposite.

A series of new polyurethane–clay hybrid materials was successfully prepared. The prepared hybrid nanocomposites were homogeneous and thermally stable. The thermal properties of the polyurethane–clay nanocomposites were significantly enhanced by organoclay particles. The flame retardance, decomposition temperature and glass transition temperature of the nanocomposites increased with increasing organoclay content.

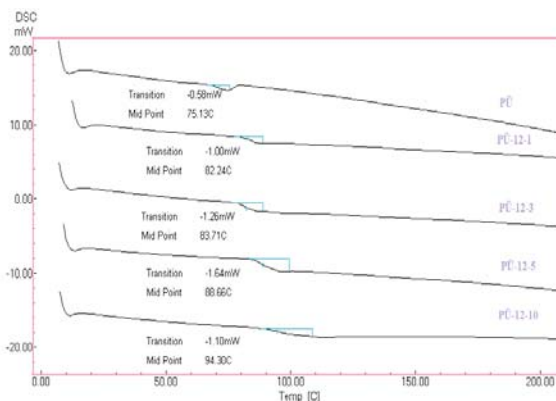


Figure 2. Glass transition graphics of PU–organoclay nanocomposite.

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Enhanced Wear Performance of SiC/Al₂O₃ Nano-Powder Reinforced Composite Coatings

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Abstract-In the present study, metallic chromium (Cr) and silicon carbide/alumina (SiC/Al₂O₃) nano powders were co-deposited via electrolytic method to obtain Cr-ceramic nano powder reinforced metal matrix composite coatings. Instead of traditional electrolytic cells, new systems were designed and coatings were fabricated in chromic acid based solutions. Low carbon steels and Pb-7wt%Sn alloy were used as cathode and anode, respectively. Before plating, substrates were polished mechanically and a serious of surface activation treatment was applied. Ceramic nano powder additional suspensions were circulated simultaneously with coating process by special apparatus. Quantitative and qualitative properties of the composite coatings were obtained using x-ray diffractometer (XRD) and energy dispersive spectroscopy attached scanning electron microscopy (SEM-EDS) machines and mechanical properties were investigated by Wear Test equipments.

To maximize the lifetime of materials, several coating techniques including vapor deposition, painting, thermal spray, galvanizing and electrodeposition can be utilized in industrial applications of these techniques *electrodeposition of metals* has special importance [1]. Traditionally, researchers have been focused on the corrosion behaviors of the zinc and chromium coatings but not on mechanical properties. It is known that not only the corrosion properties but also the mechanical properties are important for industrial applications. There are not so much alternatives to obtain materials with both improved corrosive and mechanical properties. Based on the idea determined above, *co-deposition technique* is feasible to combine the excellent corrosive protection property of Cr and both the corrosive and mechanical property of SiC and Al₂O₃.

With the shore of commentaries explained above, while some researchers focused on nickel and some other metals, and some of them focused on the static hardness and corrosion properties of these composite coatings [2], it was decided to study on the wear behaviors of them.

The aim of this study was to fabricate Cr- SiC and Cr-Al₂O₃ composite coatings with the aid of stirrer pump, magnetic stirrer and air ventilation to suspense the ceramic particles in the electrolyte and consequently to characterize the coating morphologies and investigate the mechanical properties. In this context, the coatings were by electrodeposition technique. The produced coatings were characterized by X-ray diffractometer (XRD), scanning electron microscope (SEM) including energy dispersive spectroscopy (EDS). Mechanical properties of the coatings were examined by Wear Test Machine (*ball-on-plate mode*) and Optic Microscope results to expose ceramic particle effect on mechanical properties in same conditions. For this set of the study, three different electrolytes (bath) were described for the composite electrodeposition named, *Bath-Ref.*, *Bath-S (SiC additional)* and *Bath-A (Al₂O₃ additional)*. To avoid the precipitation of the ceramic content in the electrolyte, the baths were circulated with magnetic stirrer, air ventilator or both magnetic stirrer and air ventilators.

From the map analyze results of the samples (Figure 1) fabricated in electrolyte S1 and A1, it was obtained that nano silicon carbide and alumina ceramic powders were co-deposited successfully.

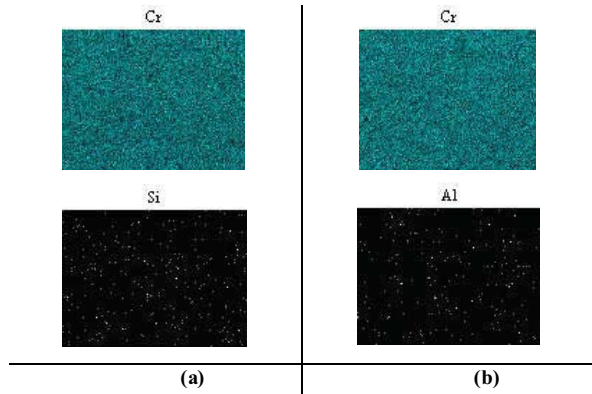


Figure 1. Map analyze results of co-deposited Cr-SiC and Cr-Al₂O₃ coatings, respectively.

Table 1. Optic images of the ball samples after wear test and wear efficiency according to depth.

	Optic Image	Wear Efficiency according to depth (nm)
Ref.		200
S1		155
A1		180

Table 1 shows the optic images of the ball samples after wear test. It can be clearly seen that S1 and A1 coded samples show enhanced wear efficiency compared to reference coating. % wear efficiency values were found to be 22.5 and 10 for Cr-SiC and Cr-Al₂O₃ composite coatings, respectively.

The study was supported by Ministry of Industry and Trade with the project code 0099-STZ-2007-1.

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Thermal Characterization of Nano Structural Metal Ion Composites via Direct Pyrolysis Mass Spectrometry

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Abstract- In this work nanostructural Co²⁺, Cu²⁺ and Cr³⁺ functional poly(vinyl pyridine)-block-poly(methyl methacrylate) (P2VP-b-PMMA) composites were prepared and characterized by classical techniques such as TEM, ATR-FT-IR and UV-vis spectrometry. Studies with the use of direct pyrolysis mass spectrometry indicated coordination of metal ions to both blocks through carbonyl and pyridine units of PMMA and P2VP respectively.

The synthesis of polymer composites by coordination of metals or metal ions has gained significant interest as a consequence of their uses as conductors, liquid crystals, light emitting diodes and their interesting optical, magnetic and catalytic characteristics [1, 3]. The literature work indicates that the use of block copolymers, with interesting morphologic characteristics, in the synthesis of organometallic polymers yields several advantages in the preparation of nano structural metal composites. Though several studies on preparation and application of these important materials have been carried out, the knowledge of thermal characteristics that are very important for application areas is still limited [4, 5]. Application of TGA for the purpose of determination of thermal characteristics is very common, yet, the results obtained are insufficient for investigation of thermal degradation mechanism and products.

In this work, synthesis and characterization of nano structural metal ion composites and investigation of thermal characteristics have been aimed.

In the first part of this work, coordinations of Co²⁺, Cu²⁺ and Cr³⁺ to poly(vinyl pyridine)-block-poly(methyl methacrylate), P2VP-b-PMMA, were achieved according to the literature methods. For this purpose, the solutions obtained by adding CrCl₃.6H₂O, CoCl₂.6H₂O, or CuCl₂ samples to toluene solution of P2VP-b-PMMA were refluxed at 110 °C for about 8hrs. Upon evaporation of the solvent solid products were characterized via classical techniques such as TEM, ATR-FT-IR, UV-vis spectrometry. The TEM images proved the formation of nanoparticles. The disappearance of characteristic peaks due to pyridine stretching and bending modes in the FTIR spectra of the samples confirmed the coordination of metal ions to the pyridine nitrogen. Furthermore, the peak due to CO stretching of PMMA decreased in intensity while a new absorption peak appeared in the FTIR spectra pointing out that electron deficient metal ion also coordinated to carbonyl oxygen in order to compensate its electron deficiency.

In the second part of the study, thermal characterization of the samples was achieved via direct pyrolysis mass spectrometry. The results indicated an increase in thermal stability of both components. The single ion pyrograms, (the variation of ion yield of a thermal degradation product as a function of temperature) of methyl methacrylate monomer ion and dimer of 2-VP during the pyrolysis of P2VP-b-PMMA, and Cr³⁺ coordinated P2VP-b-PMMA are shown in Figure 1 and 2 respectively as an example. Drastic increase in the thermal stability of both homopolymers was detected upon coordination to Cr³⁺. However, the effect was more dominant for the PMMA block. Significant changes in the relative yields of thermal degradation products were detected indicating that not only thermal stability but also thermal degradation mechanisms of both components have changed. Similar results were obtained when Cu²⁺ and Co²⁺ were used.

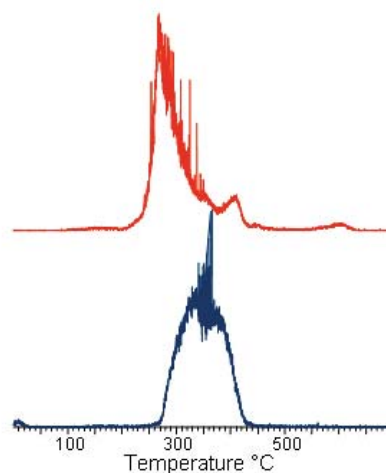


Figure 1. Evolution profiles of MMA monomer during the pyrolysis of P2VP-b-PMMA, and Cr³⁺ coordinated P2VP-b-PMMA

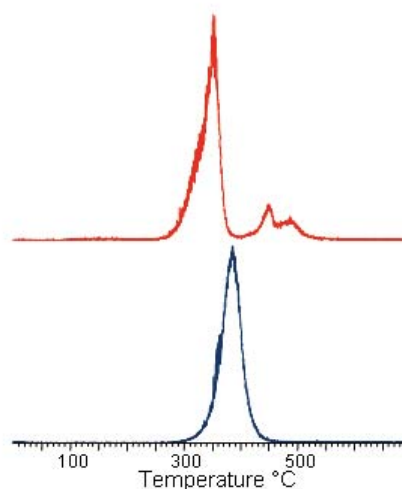


Figure 2. Evolution profiles of 2-VP dimer during the pyrolysis of P2VP-b-PMMA, and Cr³⁺ coordinated P2VP-b-PMMA

This work was partially supported by TUBITAK under Grant No. TBA G-104T536.

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Effect of Synthesis Variables on the Fluorescence Properties of CdSe-Polystyrene Nanocomposites

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Abstract-We performed the synthesis of CdSe QDs-polystyrene nanocomposite by miniemulsión polymerization varying surfactant and initiator levels to assess their effect on the material photoluminescent properties. Latexes showed stable luminescent emission as a function of polymer particle size, which was determined by the amount of surfactant and initiator used in the synthesis process.

Quantum dots (QDs) encapsulation in a polystyrene matrix allows obtaining polymeric photoluminescent nanocomposites. The luminescence properties of QDs depend largely on the environment in which they are contained [1]. That is why in addition to providing support to the nanocrystal array that maintains the photoluminescent effect of the material must also ensure a process of synthesis that does not affect the luminescent properties of the QDs.

The miniemulsión polymerization process is the formation of monomer droplets uniformly dispersed and stable within a continuous phase and later became small reactors that can produce a latex polymer with characteristics determined by variable kinetics and synthesis of the same [2,3]. This synthesis technique allows the encapsulation of CdSe nanocrystals in the monomer drops since the beginning of the process by avoiding diffusion processes that affect their structural characteristics and emission [4,5].

The synthesis processes were conducted varying levels of surfactant concentration and initiator. Cetyl trimethylammonium bromide (CTAB) was used as the colloidal stabilizer, its critical micelle concentration was determined by electrical conductivity in a system of water-styrene miniemulsión establishing a minimum concentration of $1.6 \times 10^{-3} M$. Two 2,2-azobisisobutyronitrile (AIBN) initiator concentrations, 0.5% and 0.75 wt% in respect to monomer content were established to assess their effect on the fluorescent emission of compounds. The amount of QDs applied to each treatment was 0.15 wt%. Polymerization conversions were gravimetrically measured. The average particle size, particle size distribution and polydispersity index of polymer particle was determined by statistic method considering the measurement of at least 500 particles. Composite micrographs were obtained by a field emission electron microscope Jeol JSM-7401F. Photoluminescence (PL) spectra of CdSe/PS composite were acquired using a Varian Eclipse spectrofluorometer at 360nm excitation.

Miniemulsión polymerization process was achieved at 70 °C with continuous magnetic stirring and nitrogen atmosphere. The latex obtained are stable and showed fluorescence by excitation with UV light, indicating that the method used prevented QDs deactivation also known as quenching (Fig. 1).

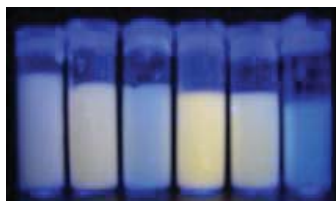


Figure 1. Luminescent emission of CdSe-polystyrene nanocomposites with variation in levels of surfactant and initiator during the miniemulsion polymerization synthesis.

The increment in surfactant level reflected a decrease in the polymer particle size, which increased in turn rate of polymerization (0.0203mol/Lmin) and percentage conversion to values of 99.8%. On the other hand, the effect of varying the initiator concentration caused the rise in the rate of polymerization, however, not significantly affected the particle size of polymer particles obtained. The fluorescent emission of the latexes varied as a consequence of variations on both surfactant and initiator concentration (Fig. 2).

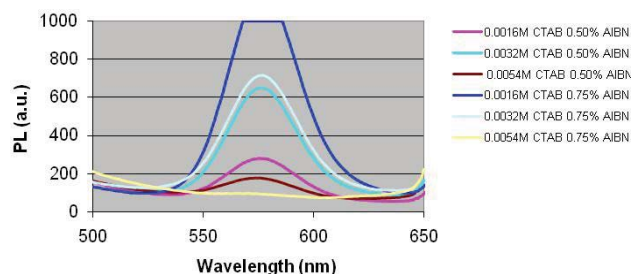


Figure 2. Photoluminescent emission spectrum of CdSe-polystyrene nanocomposites.

A feature of the emission spectrum obtained was that all samples have a relatively narrow emission profile, this condition can be indicative of low polydispersity in polymer particles [6]. Furthermore, the position of maximum excitation energy between the different samples showed no changes from an excellent particle size distribution with a strong quantum confinement [7].

The emission results obtained revealed a successful encapsulation of nanoparticles in the polymer matrix and the intensity photoluminescent properties of the nanocomposites undergo changes due mostly to the changes in the polymer particle size, which were induced by variations in levels of surfactant and initiator used.

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Preparation of Nanoceramic La-doped Bi₂O₃

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Abstract—Electrospinning process was utilized to fabricate La-doped Bi₂O₃ nanoceramic composite for SOFC applications. PVA was used as polymer solution with bismuth acetate/lantanium acetate as precursor material, followed by calcination of electrospun nanofibers at 800 °C. The produced calcined crystal powders were characterized by using SEM and XRD. The results indicated that samples after calcination have nanosized homogeneous spherical grains with 120 nm size. The XRD pattern Bi₂O₃-La₂O₃ powder sample exhibits metastable β phase.

During the last decades, solid oxide fuel cells (SOFC) represent a promising alternative for large scale electricity generation. There has been considerable interest in Bi₂O₃ and its compounds for SOFC applications in recent years due to the high oxide ion conductivity observed in these systems [1]. Various methods were used to prepare Bi₂O₃ such as mechanical grinding and mixing of related oxides or spark plasma sintering technology but both methods result a lower thermoelectric performance. Recently, wet ceramic techniques such as sol-gel are favored due not only about the cost aspects [2] but also preparation of ultra fine and ultra homogenous microstructures [3].

In this study, composite polymer solutions were electrospun to obtain ultrahomogenous and nanosized crystal structures. In the literature, electrospinning technology has been applied to prepare ceramics, and polymer/ceramic composites [4]. Most of the solid solutions of Bi₂O₃-M₂O₃ (where M is rare earth oxide such as Ca, Sr, Y, Ba, La, Gd), that are used as ionic conductors. The purpose of this work was to synthesize and characterize Bi₂O₃-La₂O₃ samples. This solid solutions were prepared because of their crystal structure is rhombohedral and it is found that the rhombohedral phase is formed in the case of relatively large M³⁺ ion [5]. In the experiments, PVA with a molecular weight of 72000 g/mol was used and bismuth(III) acetate and lantanium(III) acetate were obtained from Sigma Aldrich and deionized water was used as a solvent. Aqueous PVA solution (10%) was first prepared by dissolving PVA powder in distilled water and heating at 80 °C with stirring for 3 h, then cooling to room temperature. 1 g of the bismuth(III) acetate and 0,27 g of lantanium(III) acetate were added to the 100 g aqueous PVA at 60 °C separately and drop by drop and the solution was vigorously stirred for one hour at this temperature. Thus, a viscous gel of PVA/Bi-La acetate solution was obtained. The composite polymer solution was poured in a syringe, the needle being connected to the positive terminal of a high-voltage supply able to generate DC voltages up to 40 kV. The suspension was delivered to the needle by a syringe pump (New Era Pump Systems Inc., USA). The distance between the tip of the needle and the aluminium collector was fixed at 18 cm. The following operative parameters were chosen: flow rate 0.5 ml/h, applied voltage 18 kV. Nanofiber mats were calcined at 800 °C at atmospheric conditions for 2 hrs. Fiber morphology were determined by SEM (JEOL JSM (5410 Lv) on samples sputtered with gold. Figure 1. a) shows SEM micrograph of electrospun nanofibers. Fiber diameters were quantitatively measured using ImageJ software. The average fiber diameter for electrospun PVA/(Bi/La) acetate nanofibers were 449 nm. The SEM image shows that the nanofibers have linear, smooth and

uniform structures with no beadings. Figure 1. b) shows SEM micrograph of La-doped Bi₂O₃ crystal structures with uniform spherical grains. Grain diameters are also measured using ImageJ software as 120 nm. Grain size is very important for crystal structure because at high operation temperatures, high grain size cause cracks.

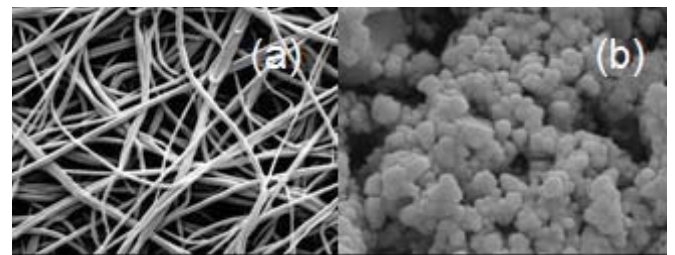


Figure 1. SEM micrographs of a) electrospun nanofibers. b) La-doped Bi₂O₃ calcined at 800 °C

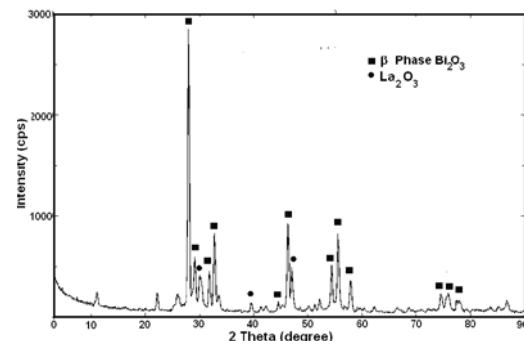


Figure 2. XRD pattern Bi₂O₃-La₂O₃ powder samples

The XRD pattern Bi₂O₃-La₂O₃ powder sample exhibits metastable β phase (JCPDS 76-147) as can be seen from the spectra (Fig. 2) with characteristic 2θ values at 27.66° (201), 29.96° (211), 31.11° (002), 32.75° (220), 45.88° (222), 47.03° (400), 53.42° (203), 55.39° (421), 57.36° (402), 73.77° (423), 74.76° (224), 75.74° (601), respectively.

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Carbon nanotube (CNT) Reinforced Alumina Based Ceramic Nanocomposites by means of Hydrothermal Synthesis and Spark Plasma Sintering

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Abstract- Multiwall carbon nanotube (MWCNTs) reinforced nanocrystalline boehmite nanocomposite powders were synthesized by hydrothermal synthesis (HS) using aluminum acetate as starting material mixed with CNTs prior HS. CNT distribution in the matrix and surface functionalization of CNTs were achieved via HS. The particles of boehmite consists of cubic crystals averaging 40nm in length. Obtained composites powders were densified using spark plasma sintering (SPS) at 1600°C to obtain CNT/alumina composites with near full density. The effect of CNTs on particle/grain size and conductivity of alumina was evaluated.

Carbon nanotubes (CNTs) have exceptional properties, such as high electrical conductivity in the range of >10⁵ S/m for multi-walled CNT (MWCNT) [1], tensile strength up to 60 GPa [2] and rigidity of the order on the order of 1TPa [3]. CNTs have great potential applications in the field of novel composites due to their very large aspect ratio (1000–10,000) and properties explained above [4]. The addition of CNTs to the brittle and insulating matrix of Al₂O₃ can increase the toughness and conductivity but the tendency of agglomeration due to the van der Waals forces between individual CNTs and achieving connections between CNT-Al₂O₃ interfaces are the main challenges posing obstacles for obtaining composites with enhanced properties [5]. Conventional powder mixing and sintering techniques are not enough to homogeneously distribute CNTs in the matrix [6]. Therefore, the treatment called functionalization have to be conducted. Functionalization is the attachment of functional groups (COOH and OH) to the sidewalls and edges of CNTs. The oxygen containing functional groups charges CNTs negatively and makes them repel each other. Conventional functionalization (3:1 H₂SO₄-HNO₃ mixture) causes surface defects on CNTs resulting in significant loss in properties [5].

In this study, a simple synthesis technique was used to produce CNT/alumina nanocomposites with enhanced properties. Aluminium acetate was used as a starting material for boehmite (γ -AlOOH) synthesis and mixed with CNTs (1wt % of the total powder) in deionised water prior to HS. Subsequently, hydrothermal synthesis was employed at 200°C for 2 h. During synthesis, boehmite formation accompanied by CNT functionalization which was achieved by functional groups obtained from boehmite formation reaction. The dried powders were densified with SPS (at 1600°C for 5 min. under 50MPa). The relative densities of obtained samples are % 99 and % 98 for Al₂O₃ and CNT/Al₂O₃, respectively. Monolithic Al₂O₃ had a DC conductivity of 10⁻⁸ S/m and the conductivity of CNT/Al₂O₃ was 10⁻⁴ S/m. An obvious improvement in electrical conductivity was achieved via 1 wt. % CNT addition. TEM and SEM analysis of SPSed

samples (Figure 1) revealed that CNTs were located on the alumina grain boundaries and there was a substantial amount of grain refinement achieved compared to monolithic alumina.

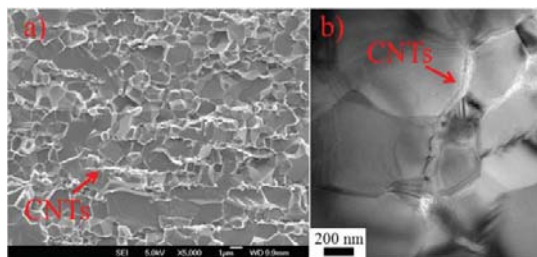


Figure 1. a) SEM and b) TEM micrographs of CNT/Al₂O₃. Grain refinement and location of CNTs between grain boundaries can be readily seen (b).

This work was funded by TÜBİTAK under contract number 108T651.

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Determination of Interatomic Forces between a Silicon Nitride Tip and α -Alumina Surface in Solution using AFM and comparison with Theory

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Abstract-Interaction forces between a silicon nitride tip and α -alumina surface were measured using AFM under different pH conditions. The force data was compared with electrophoretic measurements using the DLVO theory assuming constant potential or constant charged surfaces. In calculating the electrostatic component of the interaction, a newly developed method which is suitable for arbitrarily charged surfaces was employed. The paper includes a detailed description of the procedures used for obtaining the raw force data, its conversion to force-distance curves and comparison with the theory for various charging conditions.

Interaction forces between colloidal particles play an important role in numerous physicochemical systems in mineral, ceramic, and environmental sciences since they determine stability, rheology, and forming characteristics. Control and manipulation of these properties depend on detailed analysis of the interactions among the particles. Interparticle interactions can be divided into two main categories; van der Waals (vdW) and Electrical Double Layer [1,2].

Van der Waals forces develop between any two macroscopic particles due to the presence of such molecular forces as Keesom, Debye and London. In most solution conditions, they are attractive. Electrostatic forces on oxides are due to the presence of positive, negative and neutral charges which develop owing to the acid-base chemistry of the surface. Electrophoretic potential measurements are widely employed to obtain a measure of the degree of charging of such surfaces.

Atomic Force Microscopy (AFM), which is usually employed to obtain a topographic "picture" of the surface, allows measurement of these forces between surfaces [3,4]. What is measured with an AFM is a net force which is a combination of Van der Waals and Electrostatic Interactions for well defined solution chemistry.

In this study, systematic measurements of the interaction forces between a silicon nitride probe and an α -alumina substrate were carried out using the AFM as a function of pH and compared with the DLVO theory. The paper includes a detailed description of how such force measurements can be carried out (surface preparation, surface conditioning, statistical accuracy etc.) and how the raw data can be converted to force-distance information (determination of the spring constant, setting up the algorithm for the conversion, etc.) and how the force-distance information can be compared with the theory. The study also includes the first use of a newly developed analytical calculation method [5] for the electrostatic component for any charging condition which could only be computed using numerical methods up to now. The specific purpose in this part was to have a feeling of the extent of the range in which this theory was applicable.

A planar α -alumina sample (1x1 cm from MTI, CA, USA) was used as the substrate in all tests. Rectangular silicon nitride cantilevers (Type ORC-10, Veeco Instrument, CA, USA) employed as the probe. The cantilevers spring constants were determined before each AFM force measurement. The degree of surface charging for both materials was determined by zeta potential measurements of SiO_2 and Si_3N_4 powders at various ionic strengths (Figure 1).

Though the AFM force measurements between α -alumina substrate and Si_3N_4 tip were carried out at various ionic

strength and pH, the results presented in Figure 2 are only for pH 3 at 10^{-3}M KCl.

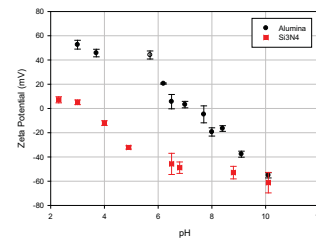


Figure 1. Zeta potential of α -alumina and silicon nitride powders at 10^{-3}M KCl.

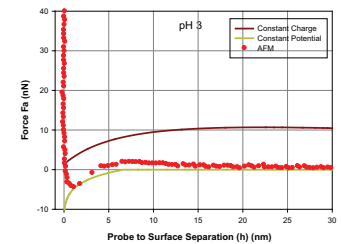


Figure 2. Effect of solution pH on the measured and theoretical interaction forces between α -alumina substrate and Si_3N_4 tip.

Interaction forces between a silicon nitride tip and α -alumina surface were measured using AFM at pH 3,7,10. The force data was compared with electrophoretic measurements using the DLVO theory. In calculating the electrostatic component of the interaction, a newly developed method which is suitable for arbitrarily charged surfaces was employed.

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