Objectives and activities - Period I-2013

Objective 1. Design conceptual models for composite inorganic particle – polymer coatings with nano- micro-patterned surface

1.1. Analysis and evaluation of the preparation methods for obtaining iron oxide nanoparticles with hydrophobic surface. Formulation design.

1.2. Analysis and evaluation of the preparation methods for the polymeric matrix. Formulation design.

1.3. Analysis and evaluation of the preparation methods for composite coatings. Formulation design

Conclusions

The study of the current state of the art in the field led to proposing the following strategy for the composite material synthesis:

- The iron oxide nanoparticles will be obtained by wet synthesis methods, namely co-precipitation or partial oxidation, followed by functionalization with surfactants with variable hydrophobicity; the formulation will be optimized to yield nanoparticles able to magnetically assemble during the curing stage of a polymeric matrix to create coatings with controlled roughness;
- The polymeric matrix will consist in acrylic copolymers or chitosan grafted with acrylic monomers, subsequently crosslinked;
- Model films will be prepared by spraying or by spin coating;
- The surface roughness will be generated by nanoparticle aggregation in magnetic field.

Objectives and activities - Period II-2014

Objective 1. Studies regarding iron oxide particle preparation, evaluation/properties optimization

1.1. Iron oxide nanoparticle synthesis

1.2. Nanoparticle characterization regarding size distribution, magnetic properties and colloidal stability in hydrophobic solvents

1.3. Experimental evaluation of the synthesis parameters. Optimum formulation selection.

Conclusions

Magnetite nanoparticles bearing variable hydrophobic functionality were prepared by co-precipitation and partial oxidation method, followed by surfactant coating.

While both synthesis methods yielded well crystallized, uniformly sized magnetite nanoparticles that were able to generate patterned composite films under the influence of magnetic field, the product obtained by partial oxidation proved more suitable for the envisioned application due to:

- higher phase purity and saturation magnetization;
- better surfactant coverage;
- ability to produce films in which the degree of aggregation and patterning may be controlled by the surfactant nature or by the magnetic field strength;
- coatings with hierarchical roughness, consisting in micro-rods with nano-scale structuring that may facilitate air pocket entrapment beneath water drops.

The results show that textured surface composite coatings may be prepared in a relatively simple manner with the aid of a magnetic field. Moreover, the versatility of the method is demonstrated by the possibility to choose the surfactant combination with the appropriate HLB value, depending on the support properties, to encourage the desired surface patterning.

Future work will be carried out for synthesizing a polymeric matrix modified with a low surface energy reagent in order to produce composite films with ice-phobic properties.

Objective 2. Studies regarding polymeric matrix preparation, evaluation/ properties optimization

2.1. Polymeric matrix synthesis by solution polymerization

2.2. Selection of co-monomers and initiator

2.3. Polymeric matrix characterization regarding chemical composition (FTIR, NMR) and molecular weight distribution (GPC)

2.4. Experimental evaluation of the synthesis parameters. Selection of the optimum formulation.

Conclusions

- A method for grafting acrylic monomers onto chitosan by solution polymerization was proposed and optimized. The results show that the mixtures of butyl acrylate or methacrylate with ethylene glycol dimethacrylate showed the highest grafting conversions;
- The grafted chains are short and dense. The obtained copolymers are soluble in water;
- The model films prepared with grafted copolymers are hydrophilic;
- A method for sol-gel crosslinking of chitosan or its copolymers with silane was proposed and optimized. The model films prepared with chitosan-silane hybrid materials are hydrophobic.