Sustainpack

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Instrument: **IP**

Thematic Priority

Deliverable 3.08

"REPORT ABOUT METHODS TO EVALUATE MECHANICAL, OPTICAL, THERMAL AND BARRIER PROPERTIES"

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Revision 1

Nanofibres/nanoparticles and matrices of interest in this project for the development of sustainable nanocomposites materials have been defined after the first six months of work (see Deliverable 3.04) and several different systems have been selected at this stage. In view of the number of nanoparticle/matrix pairs to be studied (larger than twenty), a systematic detailed program of property characterization for all materials cannot be imagined. Nevertheless, results from the developments of all systems considered will be investigated according to a general common approach which includes different levels of characterization, which are briefly described hereafter.

a) A preliminary characterization will be performed on all nanocomposite systems, devoted to investigation of material structure and specifically to the evaluation of the degree of dispersion of nanoparticles in the host matrix. Indeed, it is well known that significant improvement of mechanical and gas barrier properties can only been obtained if interface area between nanometric inclusions and polymer host matrix is maximized. In this respect, optimal results in terms of material properties are expected from nanoclay/polymer systems for those cases in which "exfoliation" of original clay particles is obtained. This first characterization activity will allow to recognize those systems and preparation protocols which are expected to offer better results. On the basis of analysis of these data, best systems will be selected for the direct measurement of relevant material properties.
b) For the case of systems in which a convenient dispersion of nanoclays-nanofibres in polymeric host matrix has been reached, a specific characterization activity is scheduled aimed to the evaluation of mechanical and optical properties, as well as gas barrier characteristics. Results from this experimental activity will allow to directly evaluate the effect of nanoparticles inclusion on the properties of polymer matrix and comparison of data obtained from different process conditions will be used to provide valuable feedback information for the proper tuning of the process parameters. The latter represent a key result for the industrial implementation of the process, scheduled at a later stage in this subproject.
c) Nanocomposite systems which proved to offer interesting results in terms of mechanical, optical and gas barrier properties will be finally analysed in order to evaluate component migration in food contact applications. This characterization activity is aimed to prove that systems developed in the subproject are suitable for the production of new materials for food packaging applications.

A list have been prepared of all method and instruments available to partners in this subproject which can be useful for the preliminary characterization of material structure, for the evaluation of mechanical, gas barrier and optical properties as well as to perform tests for food contact applications. The overall picture obtained through this analysis is reassuring indeed, as it results that the overall consortium of partners is well equipped for all the characterization activity scheduled in this work package. Indeed, a program has been set for the characterization of material under developments, in terms described above, in which activity of experimental analysis of the various systems have been distributed among all partners involved in the subproject. On the other hand, in view of the large number of systems to be investigated, different experimental resources need to be used for different systems and a unique set of experimental tests to perform on material samples for each characterization activity cannot be easily determined. In general terms, it can be said that preliminary characterization will take advantage of analyses included in following list: Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analysis (DMTA), Thermal Gravimetric Analysis (TGA), Wide Angle X-ray Scattering (WAXS), Small Angle X-ray Scattering (SAXS), Infra Red Spectroscopy (IR), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM). From this kind of analysis, in particular, effect of process used for nanoparticle inclusion on thermal properties, degree of crystallinity and thermal/mechanical transitions in polymeric materials will be examined. Degree of nanoparticle dispersion will be specifically investigated through WAXS and TEM analysis. More specific analysis of material structure could be performed for the case of selected systems, taking advantage of the facilities available to some of partners in consortium: Size Exclusion Chromatography (SEC), Attenuated Total Reflectance Fourier Spectroscopy (ATR/FTIR), Raman Spectroscopy, Small-angle neutron scattering (SAN), Field Emission Scanning Electron Microscopy (FE-SEM).

As long as mechanical properties are concerned, information resulting from already mentioned DMTA analysis will be mainly complemented with measurement of tensile properties (tensile modulus, tensile strength, elongation at break), dart impact characteristic, static and dynamic stiffness. For a limited number of systems also effect of moisture content on mechanical properties could be examined, for its relevance in the field of food packaging application.

Gas barrier properties of materials will be examined mainly through single gas/vapour permeation experiments but also by means of single gas/vapour sorption or desorption process and mixed gas permeation experiments, at list for a limited number of systems, in order to have a comprehensive picture of mass transport properties in new nanocomposite materials (see Deliverable 3.07 for more details). Different methods will be used for the evaluation of steady state gas flux through nanocomposite films in permeation experiments: commercial apparatuses such as those in OXTRAN series, based on specific detection methods as well as in-house built systems, developed in various laboratories which participate in the project. Latter instruments, in particular, offer sufficient flexibility to use samples of different size and thickness and to measure permeability in a rather large range. Water permeation rate measurement will be also carried out in systems using gravimetric detection devices. Effect of temperature on gas permeability through nanocomposite films will be also examined for the case of selected systems, in order to evaluate the activation energy of mass transport properties. Valuable data are expected from mixed-gas sorption experiments and specifically from the case of gas permeability in samples conditioned at different moisture content. Latter kind of experimental data will be obtained by means of in-house developed apparatuses which are being currently setting up in different labs.

Effect of inclusion of nanoparticles or nanofibres on optical properties of polymer matrix will be investigated by means of gloss and scatter measurements, optical density and uniformity, brightness and whiteness, opacity and transparency. Both commercial apparatuses and in-house built image analysers will be used to this aim. Investigation about the effect of the degree of dispersion of nanoparticle inclusions on optical properties of polymer matrix is of great interest, in view of the peculiar characteristic length of dispersed phase in such systems.

For the case of test for food packaging applications, both global migration and specific migration studies. Food simulants will be used for these test as established for analysis of plastic materials. A comprehensive characterization will be obtained considering different times and temperatures in migration tests. For the case of food contact applications, organoleptic tests will be performed as well, for the case of selected systems.

It must be stressed that the first step aimed to the evaluation of the effect of inclusion of nanoparticles and nanofibres is the analysis of properties of host matrixes. It is thus scheduled for next months of the research activity the characterization of all host matrixes chosen in the subproject in terms of mechanical, mass transport and optical properties.

It is worthwhile to consider that, in view of the number of different laboratories involved in the characterization activity, using different techniques or procedure and often referring to experimental conditions slightly different, the comparative analysis of results obtained by different laboratories for the exam of the same reference material is in order. To this aim, PLA samples provided by the same commercial supplier will be used by all laboratories for a standard characterization of tensile/stiffness, gas/vapour permeability and optical properties. Migration tests will be also performed on the same material. Also characterization of basic morphological properties of the same PLA material is scheduled in the same program. Results from this characterization activity not only will represent a meaningful comparison between results obtained by different laboratories but it will also provide the value of relevant material properties for a specific product currently used for packaging films. The latter values will serve as a baseline for the evaluation of the same properties in the new nanostructured materials to be developed in this project.