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Short Term Scientific Mission Report

Reference: COST Action FP1405 (ActInPact)
STSM Title: Rheology of functional materials for smart packaging
STSM Dates: 19-30th September 2016
Host Institution: Welsh Centre for printing and coatings (WCPC), Swansea (UK), Prof. Dr. Tim Claypole

The STSM at Welsh Centre for printing and coatings (WCPC) at Swansea was accomplished according to the previously prepared working plan. The researchers at the WCPC presented the use of various laboratory equipment like printing equipment (screen printing, pad printing, flexo printing…), rheological characterization (Malvern, Bohlin), contact angle/surface tension measurements, optical microscopy, tack tester, standard drying analyser... In the field of rheology, the measurements are performed mainly at Bohlin rheometer – shear/shear oscillatory and Malvern rheometer – Kinexus with advances in shear and vertical (axial) testing. The visit of the laboratories was followed by a detailed discussion about the experimental methods and tests, which could be used for various materials, prepared at National Institute of Chemistry (NIC) and were brought to Swansea. In the following days several experiments on the real samples from Slovenia were conducted with the help of researchers of the WCPC.

Sol-gel rheological characterization was performed with photochromic functional sols. The sols were used for sol-gel characterization and determination of sol-gel point with Controlled Stress Parallel Superposition (CSPS). This technique was studied in details by James Claypole in his PhD thesis (Application of advanced rheometric techniques to printing fluids, Swansea University, 2015) from WCPC, who helped with the experiments. The rheometric CSPS studies use controlled stress parallel superposition (CSPS) in probing microstructural changes that occur in response to a unidirectional shear stress applied to a material undergoing a sol-gel transition. The sol-gel transition is monitored through the use of a repeated sequence of oscillatory waveforms superimposed upon a constant unidirectional stress component, each waveform within the sequence being characterised by a discrete frequency. Identification of the gel point is hence possible by locating the point at which δ (tanδ=$G''G'$) is frequency independent. The sols, prepared in Slovenia, exhibited strong temperature dependent sol-gel transition, therefore the experiments were performed at different temperatures (20°C, 30°C, 40°C, 50°C, 60°C). Two sols on the base of WO_3 were used for the same type of experiments for the determination of the sol-gel point with rheometric CSPS studies. Gel strengths were determined for the all gels, obtained by gelation at various temperatures. After the experiments were finished all the data were collected and were evaluated with the computer programs. The results showed that the technique, used for

the determination of the gel-point, developed at WCPC, enables a clearer determination of the gel-point and characteristics of the microstructure in the near vicinity of the sol-gel transition. The results, obtained with this technique enable the calculation of the gel strength of the material. The technique could be applied to several materials, gelling and non-gelling, prepared in the Department for Materials Chemistry at NIC in Slovenia.

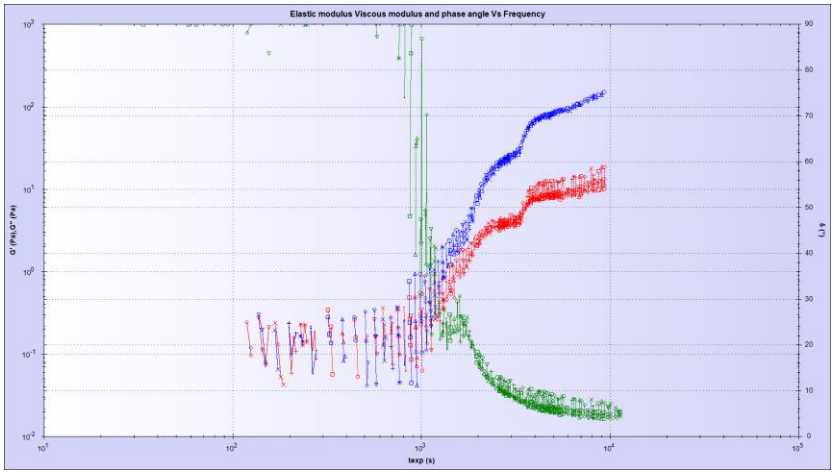

Figure 1: Experimental results of controlled stress parallel superposition (CSPS) for WO_3 sol at 40°C.

Rheological characterization of suspensions was performed with suspensions consisting of conductive nanoparticles dispersed in a fluid medium (**Figure 2**). Various rheological tests were used in order to determine the rheological properties of the suspensions and some new methods were tested with these samples.

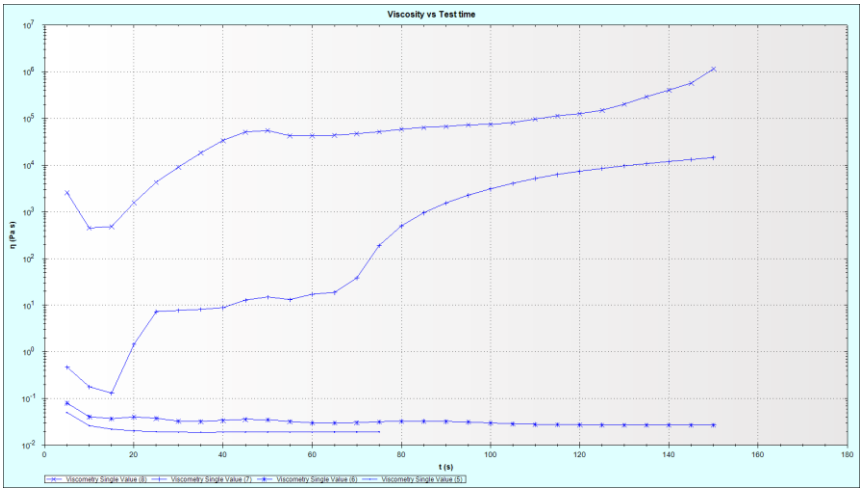

Figure 2: Experimental determination of yield stress value for the suspension of conductive nanoparticles.

Rheological characterization (temperature tests) of thermochromic materials were performed with similar experimental tests as were used for sol-gel determination since these materials go through the phase change as the temperature changes. The test enabled a detailed characterization of phase transition, but the instrument was not able to cool the sample to temperature low enough to start the phase and colour change. The method will therefore be used on these materials in Slovenia, where the rheometer Physica MCR301 enables the temperature control down to -20°C.

Printing of the photochromic (WO_3) sols was conducted with various printing techniques. Flexo printing was performed with IGT Reprotest Flexography (**Figure 3**) with different printing patterns. The same sol was used also for the printing with pad printing technique with TPX300 (Teca Print) with open inking system.

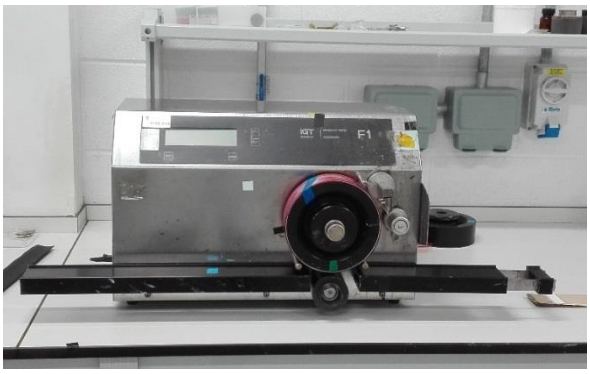

Figure 3: IGT Flexographic Printability Tester F1.

Measurements of **drying of the applied ink** (**Figure 4**), printed on the substrate were performed on the HORUS FORMULACTION Advanced Drying Analysis, which enables the monitoring of microstructure changes during the film formation process.

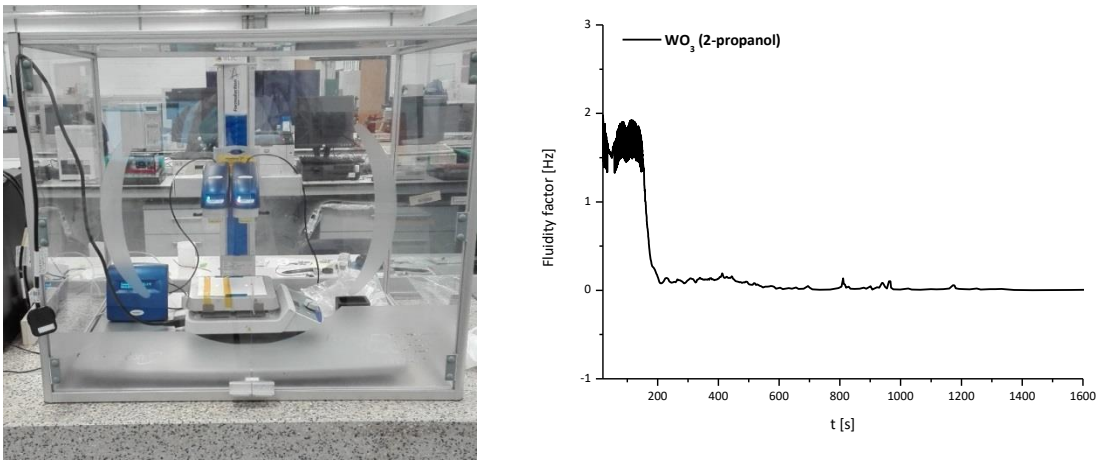

Figure 4: Measurements of drying process for WO_3 sol at HORUS FORMULACTION Advanced Drying Analysis.

The surface of the samples, applied on the glass substrates, was characterized by the White light interferometric profilometry with Wyko NT9300 (**Figure 5**). The instrument uses while light interferometry to produce a topographical image of the sample under study allowing the operator to measure a number of different parameters. The roughness can then be measured along any line in the x or y axis of the resultant image. The 2D parameters were used as they are better understood than 3D surface parameters as the ISO standard relating to the measurement of 3D surface parameters is yet to be published.

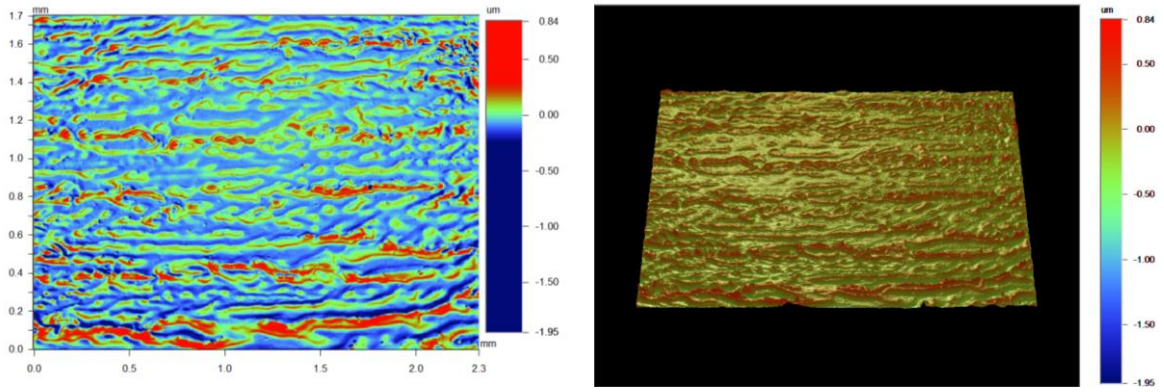

Figure 5: Plast na steklu, natisnjena z WO_3 solom in fleksotiskarsko tehniko tiska, posnetka zajeta z White light interferometrom (Wyko NT9300) pri 2,7× povečavi.

During this STSM meeting the knowledge was transferred from each group and the benefits from each group were established as well. Some common interests, which were recognized during the conversations and discussions between researchers of Slovenian and UK group, could result in new collaborations between UK and Slovenia.

Acknowledgements

I would like to greatly thank everyone at the Welsh Centre for printing and coatings (WCPC) at Swansea, especially dr. James Claypole and Dr David Beynon for all of their help and guidance with the experiments, prof. Dr Davide Deganello for the discussion and Prof. dr. Tim Claypole, who enabled the visit at WCPC.