Application Note # 135

# Gas-induced surface changes measured with MP-SPR

*MP-SPR instrument was used to characterize changes in a polymer film induced by different gases. Different humidities showed concentration dependent interaction with polymer and ethanol vapour seemed to penetrate to the polymer layer.*

## Introduction

Vapour-induced changes in material are important for several research areas e.g. environmental monitoring, pharmaceuticals (drug storing), paper or polymer research (humidity influences to material, corrosion). Especially water vapour and water soluble organic compounds are of high interest in these areas, as they can affect the lifetime of the material films, or possible products that these films are often protecting.

Excitation of the plasmons occurs at a lower angle of the incident light in gaseous environment (45°) than in aqueous environment (70°). The SPR Navi™ instruments measures a wide angular range (40-78°), which enables measurement both in liquid and in gas environment. Measurement phase is changed easily; there is no need to take sensor-slide out, change parts or recalibrate the instrument.

Vapour for MP-SPR measurement can be created with multiple ways (Figure 1): saturating gas flow by bubbling it trough liquid sample (A), using head space injections with a gas-tight syringe (B) or using mass-flow controllers connected to mixer (C).

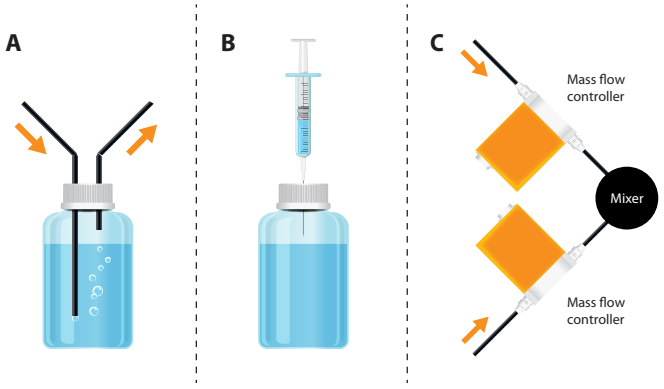


**Figure 1.** Different configurations of MP-SPR vapour measurements. A) Saturation bubbler, B) Head space injection, C) Mass flow controller and injector.

## Materials and methods

Standard SPR-Navi gold sensor-slide was coated with polystyrene – polymethylmethacrylate random co-polymer (PS-PMMA, Mw ~ 500 000 g/mol) with spin coating. 100 µl of PS-PMMA dissolved in toluene solution (5% w/w) was added on a gold sensor-slide and spinned 1 minute with 50 rounds per second speed. The goal of the coating was to form a relatively thick (hundreds of nanometers) coating, which would increase the effect of vapour if it can diffuse into the polymer.

Saturated salt solutions were prepared from potassium chloride (KCL humidity 84%) and calcium chloride ($CaCL_2$ humidity 29%) [1] in water in order to create regulated water vapour content.

SPR Navi™ 200 instrument in angular scan mode was used for measurement. Temperature of the sample was ambient 21°C of the measurement chamber was 30°C in order to avoid vapour condensation inside the flow cell, flow rate was 500 µl/min. Gaseous samples were created by saturating gas flow by bubbling it trough liquid (water, saturated salt or ethanol) sample (marked a in Figure 1). Interaction with polymer surface was measured, with ambient air acting as a baseline reference.

## Results and discussion

The coating of the sensor resulted in a 700 nm layer on the surface, and it was clearly a SPR waveguide mode –surface.

The water vapour interactions with the PS-PMMA copolymer were relatively weak. The different humidities (29%, 84% and 100%) showed a clear concentration dependent interaction with the polymer, which was characterized by following one of the SPR waveguide modes (Figure 2). The weak interaction was expected, as the water vapour does not penetrate easily into PS-PMMA polymers.

Ethanol vapour (50 and 100%) induced much larger changes than water vapour when interacting with the PS-PMMA polymer (Figure 2). This is most probably caused by the ethanol vapour also permeating slightly into the polymer surface. After the interaction the ethanol vapour also caused permanent effects to the polymer, which can be seen from the baseline ending into a lower baseline level after the interaction than what it was before interaction. This was also detected as a lowering of the refractive index of the PS-PMMA, which was characterized from the full angular curve before and after the ethanol measurement. Most probably this is caused swelling of the polymer layer upon interaction, which moves the mass further from the SPR field and lowers the refractive index of the polymer layer.

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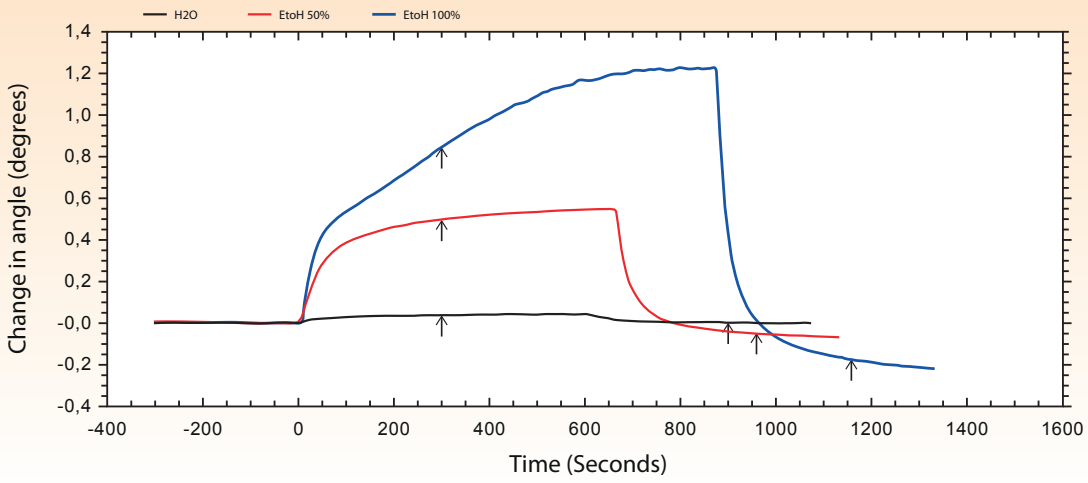


**Figure 2.** Changes in the PS surface induced by water and ethanol (50% and 100%) vapour.

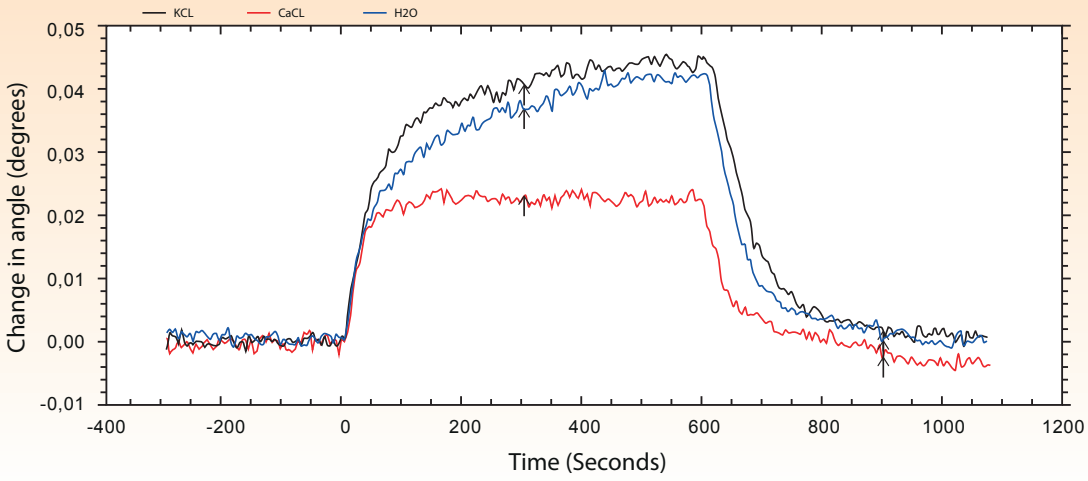


**Figure 3.** Change in the PS surface humidity induced by saturated KCL, $CaCL_2$ and water vapour.

## Conclusions

MP-SPR is excellent tool for gas-surface interaction measurements, which can be utilized in wide range of research areas such as material and environmental sciences. Gas interaction, penetration and swelling of the measured layers can be easily determined.

[1] Handbook of chemistry and physics (CRC) 94th edition 2013-2014

| **Recommended instrumentation for reference assay experiments** |
|---|
| SPR Navi™ 200 |
| Gas flow cell |
| Sensor surfaces: Au, other metal or inorganic coating |
| Spin coater, other deposition methods can be also utilized. |
| Software: SPR Navi™ Control, DataViewer, optionally TraceDrawer |

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