ESRF	**Experiment title:** Titania-containing networks for precursor materials of nanostructured carbons	**Experiment number**: SC 2918
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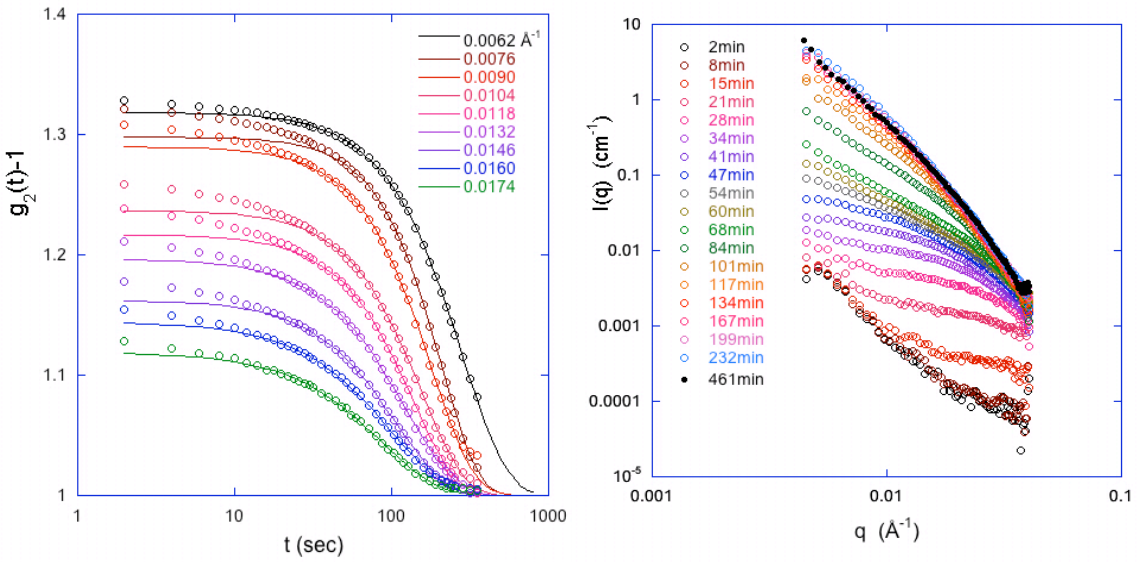
Report:

The objective of the experiment was to investigate the effect of transition metals on the gel formation mechanism and the final structure in resorcinol-formaldehyde (RF) polymer gels. The developing structure of the RF gels depends highly on initial conditions (concentration, pH, gelation temperature). Several, metal-free systems were therefore examined first in order to have information on the basic structure. It was found that the polymerization reaction at 80-90°C displays a dynamical behaviour that is too fast for the setup (i.e., using 2D detector for the XPCS measurements). In these samples only the changes in the SAXS patterns can provide information on structural development. On the contrary, at 60°C, the gelation was too slow, and again lay outside the ideal window. It was found that 70°C is the ideal temperature from both the XPCS and SAXS measurement point of view. At this temperature the polymerisation reaction exhibits non-stationary dynamics, and as the structure ages the speed of the process becomes progressively slower (Fig. 1).

The presence of a transition metal during the polymerization has a strong effect on the structure (Fig. 2a). The samples display increasingly strong surface scattering. With titania, however, the reaction rate is too fast, and only a sludge is formed. For this reason, the measurements were pursued with molybdenum (ammonium heptamolybdenate) as a catalyst. At low q the dynamics of the system in this stage remains hyperdiffusive (ballistic), as in the metal-free cases, i.e., the relaxation rate Γ is proportional to q, rather than to q^2.



(a) (b)

Figure 1: (a) correlation functions obtained during the gelation process in RF system, (b) evolution of the SAXS pattern during the gelation process

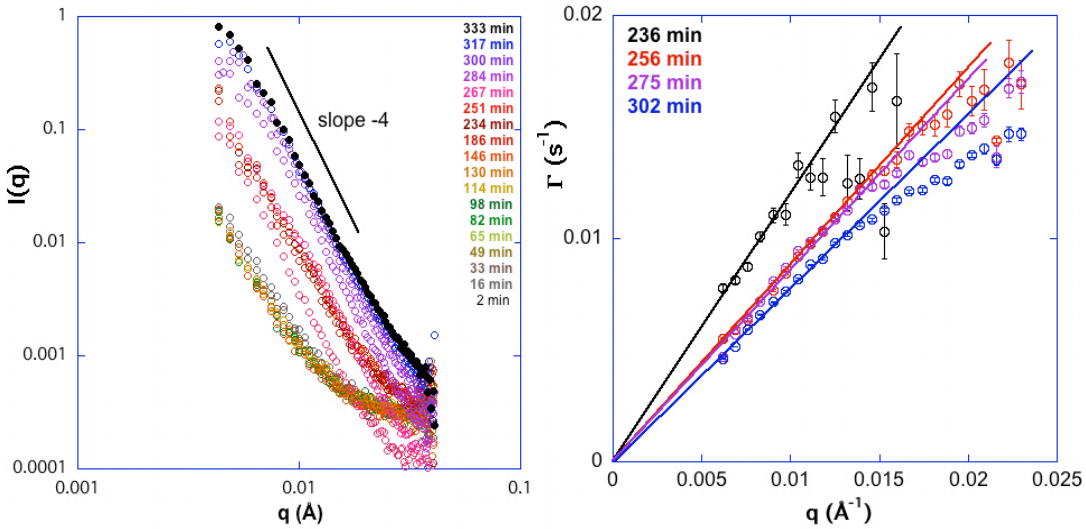


Figure 2: Evolution of the SAXS pattern (a), and variation of the relaxation rate with time (b) in molybdenum-containing RF gel system