Binary liquid mixtures separate into two phases of different compositions below a critical solution point. When imbibed in a porous matrix, the phase separation cannot occur on a macroscopic scale but the two coexisting phases form small domains and the dynamics of the growth of these domains is very slow. The interest in the structure and dynamics of phase-separated liquid mixtures in porous materials arises from the increasing importance of mesoscopically disordered systems in materials science [1], but also from applications in separation processes such as liquid chromatography, microfiltration, or the extraction of liquids from porous materials. Many of these applications depend on the flow and diffusion of the liquid in the pores, which in turn are strongly affected by the morphology of the phase separated domains.

A porous medium affects the phase separation of liquid mixture not only by geometrical confinement effects, but also by the preference of the pore surface for one component. In the one-phase region (1?) of the mixture, the latter will cause preferential adsorption, i.e., a concentration profile from the wall into the pore volume. For sufficiently wide pores, the decay length of this profile corresponds to the correlation length ? of fluctuations in the interior of the mixture, but in narrow pores (pore width D < ?) an overlap of the profiles from opposite walls will cause an enhanced adsorption. In the two-phase region (2?) the walls will be wetted, either completely or partially, by the preferred phase, causing either a tube-like or a plug-like morphology of the preferred phase in cylindrical pores [2]. Incipient wetting of the pore walls, manifested by multilayer adsorption, is expected to occur in the one-phase region close to the liquid-liquid coexistence curve in the region in which the preferred component forms the minority component of the mixture.

In order to test some of these predictions, we have studied the structure and dynamics of the binary system iso-butyric acid (iBA) + heavy water (D$_2$O) in a controlled-pore glass (CPG). The mean pore size of the CPG materials is around 10 nm. Water is preferred by the pore wall in the iBA + D$_2$O system. The phase diagram of this system in the bulk liquid state is shown in Figure 1.

![Figure 1. Bulk phase diagram of the binary liquid mixture iBA+D$_2$O. The solid line represents the binodal.](image-url)
characteristic correlation length \( \xi_D \). Figure 2 shows the temperature dependences of the correlation lengths \( (\xi_C, \xi_D) \) obtained for the 54 wt-% iBA sample.

As expected, the correlation length of the diffuse concentration fluctuations in the one-phase region increases as the phase boundary is approached. A cut off, around 8 nm, is observed: it roughly corresponds to the average pore size diameter. In the two-phases region \( (T < 30^\circ C) \), these concentration fluctuations are replaced by the microphase-separated domains of the two phases, which slightly grow in size as one moves deeper into the two-phase region [4].

The dynamics of fluctuations and microphase-separated domains has been investigated by neutron spin-echo (NSE) spectroscopy at the instrument MESS at the LLB. NSE experiments yield the time dependence of the intermediate scattering function \( S(q,t) \). They can be fitted by a single-exponential decay, \( S(q,t) \sim \exp(-Gt) \), where \( G \) is the characteristic relaxation frequency which is related to the effective diffusion constant of the domains as \( D_{et} = Gq^2 \). Figure 3 shows the temperature dependence of \( D_{et} \) for the 54 wt-% iBA-sample. The observed decrease of \( D_{et} \) with decreasing temperature is tentatively attributed to the increase of the size of the concentration fluctuations in the one-phase region and an immobilization of the domains at the pore surfaces in the low-temperature region [5].

This interpretation is consistent with the results of the SANS study, by which the increase of the concentration fluctuations with decreasing temperature in the one-phase region and the appearance of domains of the two phases at low temperatures have been directly observed. Both NSE and SANS studies show that the microphase separation of the liquid mixture in the porous matrix does not occur at a sharp temperature but extends over a wider temperature range below the phase separation temperature of the bulk mixture.

References