Experimental report

23/04/2024

Proposal:	**9-10-1764**	**Council:**	4/2023
Title:	Dynamics of Bicontinuous Microemulsions Inside Hydrophilic and Hydrophobic Confinements		
Research area:	Soft condensed matter		

This proposal is a resubmission of 9-10-1736

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Samples: controlled pore glass CPG-10
CE Bicontinuous Microemulsion

Instrument	Requested days	Allocated days	From	To
IN15	7	5	13/09/2023	18/09/2023

Abstract:

The aim of this study is to investigate the influence of a disordered mesoporous matrix on the structural properties of a complex fluid confined in this matrix. A bicontinuous microemulsion in controlled pore glasses is chosen as the model system. The average size of the oil and water domains is within the range of typical pore sizes of the confining matrix. The influence of pore sizes is investigated using two different porous matrices. In the first case, the pore sizes are larger than the domain size and in the second case, the pore sizes are smaller than the domain size. In addition, the influence of surface polarity is tested by using hydrophilic and hydrophobic pore walls as confinement. Different contrasts are used to separate the contributions of the fluctuations of the domains and the bending motions of the amphiphilic interface. The goal of this project is to understand to what extent and how the presence of a mesoscopic confinement modifies the structural and dynamic properties of the confined microemulsions, alters the phase behavior of the system under study, and possibly induces microphase separation.

Experimental Report for Proposal 9-11-1736

Performed: 13.09.2023-18.09.2023

Version: April 2024

Dynamics and Structure of Bicontinuous Microemulsion in Confinement

In this experiment, we studied the dynamics of a bicontinuous microemulsion inside disordered meso- and macroporous silica glasses (CPG – controlled pore glasses). The following materials were chosen to study the influence of:

1. Pore size – three different CPG with pore diameters of 24 nm, 46 nm, 112 nm
2. Chemical composition of the pore wall – native hydrophilic CPG surface was hydrophobically modified.
3. Bicontinuous microemulsion – two types of non-ionic bicontinuous microemulsion, a ternary system $C_{10}E_4/D_2O$/n-octane and a quaternary system Glucopon220/1-pentanol/ D_2O/n-octane

Experimental procedure

The CPG powder was pressed into a PTFE ring with a diameter of 30 mm and a thickness of 1 mm. These tablets were placed between two quartz windows in an aluminium sample holder. The liquid (D_2O, n-octane, or microemulsion) was added to the CPG tablet and covered with the quartz window. All samples were placed in an incubator at 23 °C before mounting them in the instrument.

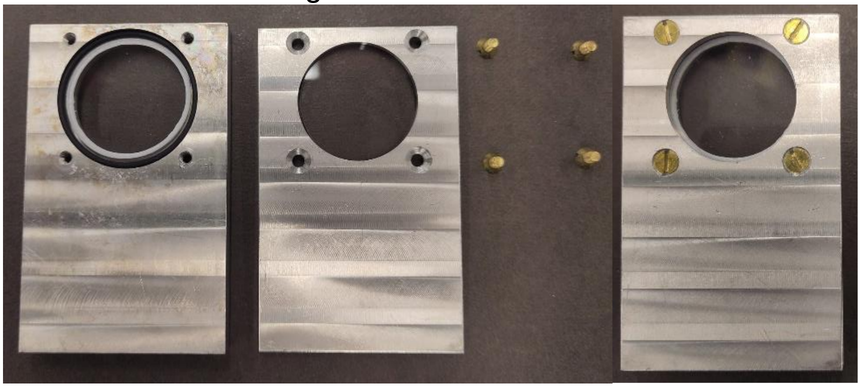

Figure 1: Aluminium sandwich cell, left: unassembled, right: assembled.

Fourier times up to 340 ns were tested at q-values between 0.05 - 0.172 $Å^{-1}$.

Table 1: Pore diameter d_P, pore volume V_P, and the porosity of the used CPGs.

CPG	d_P (nm)	V_P (mL/g)	porosity
170	24.4	0.864	0.66
500	46	1.16	0.72
1000	112	1.57	0.78

Influence of the pore size (pore diameter d_P)

The influence of the pore size of the confining matrix was studied by using CPG with different pore sizes, as listed in Table 1. The ternary system has a periodicity of $d_{TS} =$

32.7 nm, obtained from Teubner-Strey fit. Thus, the pores of the CPG170 are smaller than the periodicity and the other two CPGs have larger pore diameters.
The Glucopon microemulsion has a smaller periodicity with $d_{TS} = 10.9$ nm.

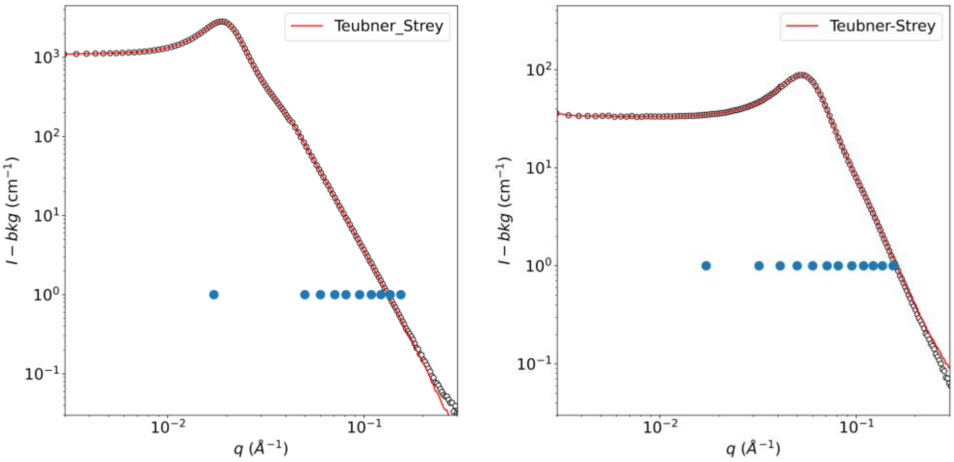

Figure 1: SANS signal of the $C_{10}E_4/D_2O$/n-octane bicontinuous microemulsion (left) and the Glucopon220/1-pentanol/ D_2O/n-octane with the Teubner-Strey fit (red line). The q-range investigated is shown as the blue dots.

For the ternary system $C_{10}E_4/D_2O$/n-octane the normalized intermediate scattering functions of the microemulsion in bulk and confined in the three CPGs are shown in Figure 2. The influence of the pore size is significant. For the larger pores (46 and 112 nm) there are still dynamics at the tested time scale observable. In the pores of 24.4 nm, there is no decay in the intermediate scattering function measured. In other words, there are no dynamics at the typical timescale for bicontinuous microemulsion observed at all tested q values. This could indicate that the microemulsion is phase-separated.

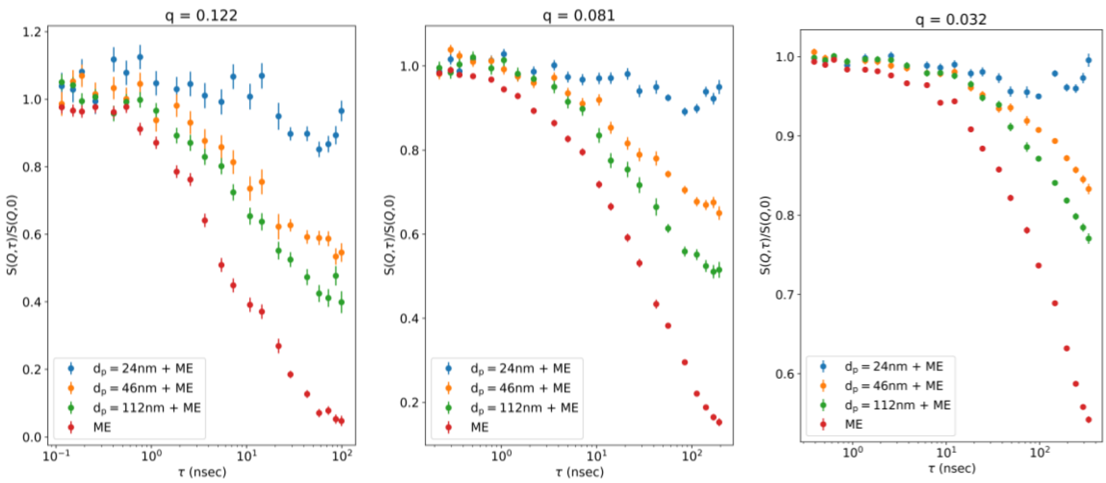

Figure 2: Normalized intermediate scattering function $S(Q,\tau)/S(Q,0)$ for the ternary system $C_{10}E_4/D_2O$/n-octane in the hydrophilic CPG with d_P = 24 nm (blue), 46 nm (orange) and 112 nm (green) for q = 0.122 Å^{-1}, 0.081 Å^{-1}, and 0.032 Å^{-1} (from left to right).

For the quaternary microemulsion Glucopon220/1-pentanol/ D_2O/n-octane, this trend was not observed, which could be because even the smallest pore size is still twice as large as the periodicity of the microemulsion.

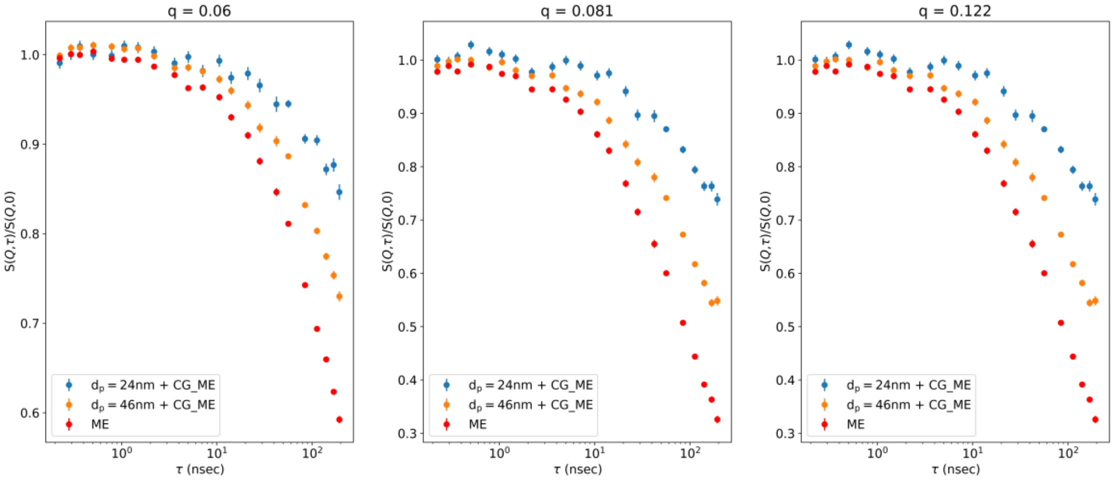

Figure 3: Normalized intermediate scattering function $S(Q,\tau)/S(Q,0)$ for the quaternary system Glucopon220/1-pentanol/ D_2O/n-octane in the hydrophilic CPG with d_P = 24 nm (blue), 46 nm (orange) for q = 0.06 Å^{-1} , 0.081 Å^{-1} , and 0.122 Å^{-1} (from left to right).

Influence of the chemical composition of the pore wall

For the ternary system, there is no difference between the microemulsion confined in hydrophilic and hydrophobic pores.

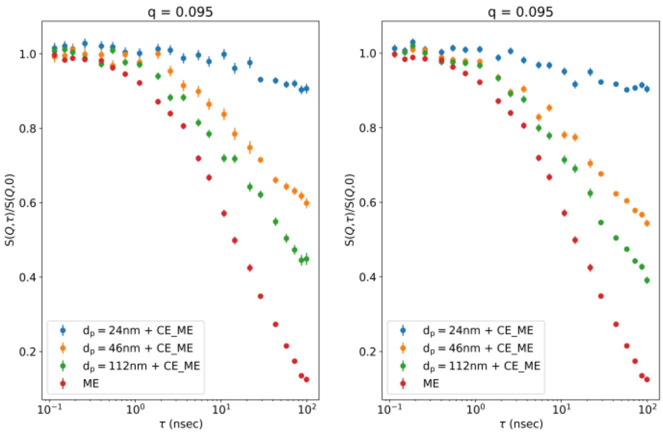

Figure 4: Normalized intermediate scattering function $S(Q,\tau)/S(Q,0)$ for the ternary system $C_{10}E_4$/D_2O/n-octane in the hydrophilic CPG (left) and hydrophobic CPG (right) with d_P = 24 nm (blue), 46 nm (orange) and 112 nm (green).

For the quaternary system, there is a slight difference in the intermediate scattering function of the confined microemulsion inside the smallest pores.

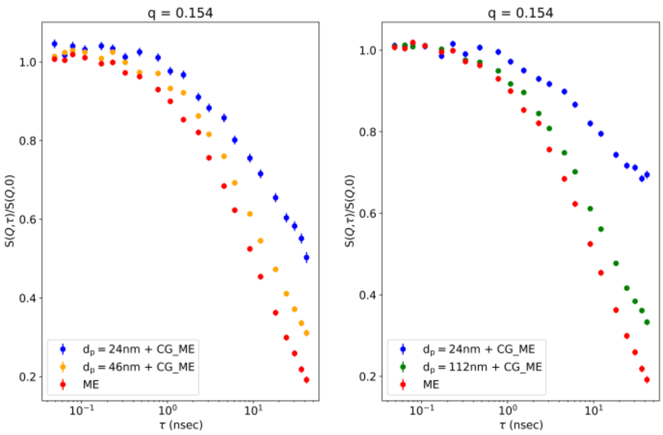

Figure 5: Normalized intermediate scattering function $S(Q,\tau)/S(Q,0)$ for the system Glucopon220/1-pentanol/ D_2O/n-octane in the hydrophilic CPG (left) and hydrophobic CPG (right) with d_P = 24 nm (blue), 46 nm (orange) and 112 nm (green).