CROSS TOKAI **Experimental Report** J-PARC	**Date of Approval:** 2013/07/12 **Approver:** J.Suzuki **Date of Report:** 2013/03/25
Project No. 2012B0126 **Title of experiment:** Effect of salt on the nanoscale structures induced in ternary microemulsions. **Name of principal investigator:** Dr. Kuperkar Ketan Chandubhai **Affiliation:** High Energy Accelerator Research Organization (KEK), Japan.	**Name of Instrument scientist:** Dr. Jun-ichi SUZUKI. **Name of Instrument/(BL No.):** BL-15 (TAIKAN). **Date of Experiment:** 27th – 30th November 2012.

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料:

No.	Name of Sample(s)	Chemical formula	Physical form	Compositions
1	Dodecyl pentaethylene glycol ether($C_{12}E_5$)	$C_{12}H_{23}(OCH_2CH_2)_5OH$	Thick, clear & transparent liquid	10% wtin D_2O
2	Sodium tetrapheny**l** borate	$NaBH_4$	Solid powder	1/10, 1/50, 1/100, 1/500.
3	Sodium dodecyl sulfate (SDS)	$NaC_{12}H_{25}SO_4$	Solid powder	1/10, 1/25
4	Bis(hexadecyldimethylammonium) ethane (16-2-16)	$((C_{16}H_{25}-(CH_3)_2)_2(CH_2)_2 . 2Br^-$	Solid powder	2.5 mM, 5 mM, 10mM
5	Bis(dodecyldimethylammonium) ethane (12-2-12)	$((C_{12}H_{25}-(CH_3)_2)_2(CH_2)_2 . 2Br^-$	Solid powder	10mM
6	Deuterium oxide	D_2O	Liquid	solvent

2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。)
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Experimental method:

Small Angle Neutron Scattering (SANS) were performed on the SANS instrument BL15 (TAIKAN) at Japan Proton Accelerator Complex (J-PARC), Tokai, JAPAN. Neutrons of wavelength λ= 0.7 – 7.6 Å were incident on samples held in a custom-built quartz banjo cell. Sample-to detector distance (5.6 m) was used to give a q - range of 0.0005 $Å^{-1} < q < 2.5 Å^{-1}$, where $q = (4\pi/\lambda) \sin(\theta/2)$ is the magnitude of the scattering vector.

Sample scattering was corrected for background and empty cell. Corrected data sets were circularly averaged.

2. 実験方法及び結果(つづき) Experimental method and results (continued)

Results and discussion:

As per the work planned in J-PARC proposal, we performed SANS experiments on 10% by weight pentaethylene glycol mono-n-dodecyl ether ($C_{12}E_5$) solution mixture in D_2O as a function of salt concentration and temperature. We have obtained some interesting SANS results and at present the fitting and analysis part is under process. Though we have presented here the SANS figures obtained after their data reduction.

As there is only little study reported on the influence of antagonistic salts on such nonionic aqueous mixture, we did select them extensively viz. sodium tetraphenylborate ($NaBPh_4$) for our work as they hold promising effect in inducing the substantial and impressive phase change even at its very low concentration. We observed the emergence of mesophases particularly multilamellar (onion) structures co-existing with the sponge textures. For such solution compositions, the prediction made on the obtained scattering profile is well evident from the polarized optical microscopy (POM) images.

Taking the reference of the phase diagram of $C_{12}E_5$ solution mixture in D_2O [Strey, R. Colloid Polym. Sci. 1994, 272, 1005], we aimed to obtain its scattering profile by SANS. Figure 1 demonstrates the temperature dependence on this surfactant mixture.

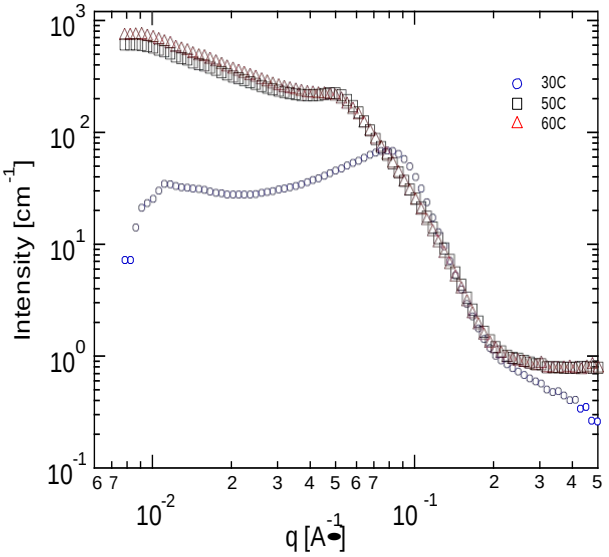

Figure 1. Measured SANS profile of 10wt% C12E5 aqueous solution at different temperatures.

As can been seen, we observed a shift in the intensity peaks (Q_{max}) towards the lower Q value on a gradual temperature increase indicating a probable structural transition from the long cylinders to high order sponges. Such typical behavior could be viewed as the thermodynamic variable which influences the microstructure transition due to the nonionic headgroup dehydration and by their efficient packing

Figure 2, explains the clear effect of the antagonistic salt $NaBPh_4$ when added to surfactant mixture. We found the peak intensity to get impressively enhanced by the gradual salt addition at high temperature predicting the homogenous distribution of the particles leading to a dramatic mesophase formation of lamellar phase (L_α) appearing to be like onion structure in coexistence with sponge phase (L_3). Such behavior could be attributed to the ion-dipole interaction between the dissociated salt ions. The scattering findings correlates with the onion shaped lamellar structures as presented in Figure 3.

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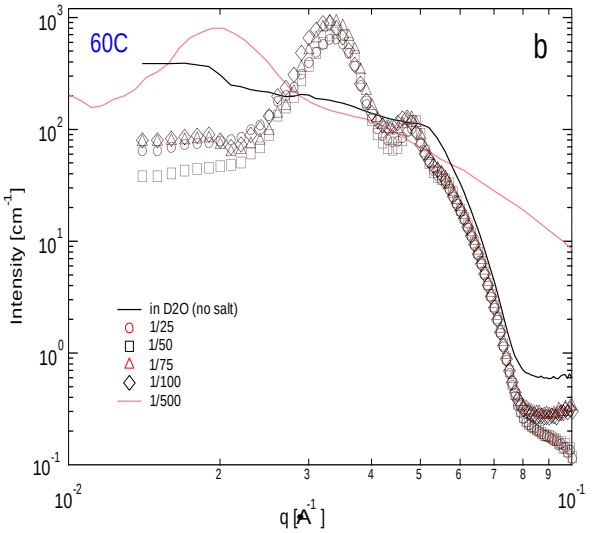

Figure 2. Temperature dependence SANS profile of 10wt% $C_{12}E_5$–D_2O mixture in different $NaBPh_4$ concentration at 60°C.

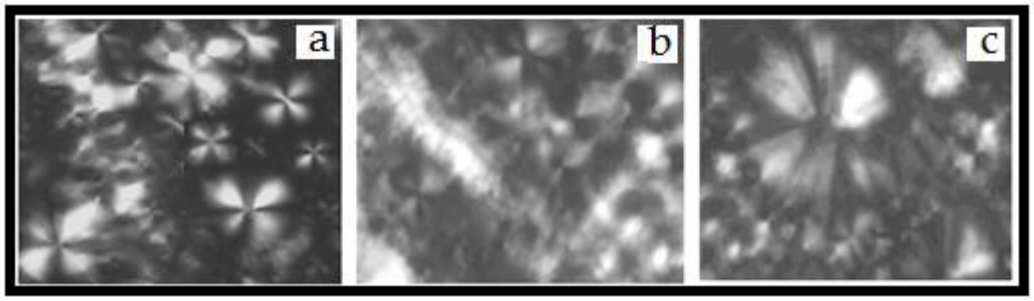

Figure 3. POM images obtained for 10wt% $C_{12}E_5$–D_2O mixture in varying [$NaBPh_4$] at 50°C. Image a show the onion structure indicating the presence of the lamellar region while image b display multi-lamellar pattern in co-existence with the sponge phase. Image (c) is the magnified view of onion arrangement.

Conclusion:

Temperature dependence phase transition study investigated the onion shaped multistructures predicting L_α phase in coexistence with L_3 for binary $C_{12}E_5/D_2O$ system at varying salt compositions. The significant change in the scattering peak intensity on the gradual salt addition with temperature increase envisaged the homogenous phase distribution which is clearer by the visual inspections obtained from POM images. However the fitted analysis parameters can throw a deep insight over this for further clear justification to our findings.

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