Probing the balance of Telechelic Polymers Bridging and Screened-Coulomb Interactions in Microemulsion System

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Abstract. The structural properties of neutral oil-in-water (O/W) microemulsions nanodroplets in the presence of telechelic polymers (PEO-2m) were studied by Small-Angle Neutron Scattering (SANS). We show that the unknown attractive interaction introduced by the addition of PEO-2m, can be balanced by a known repulsion interaction due to the addition of a cationic surfactant Cp⁺, which causes a progressive transformation of the bridges connecting the nanodroplets into loops. For this, we compute the structure factor S(q) with an effective pair potential, using the Ornstein Zernicke (OZ) integral equation approach with the Hypernetted Chain (HNC) closure relation. Generally, the agreement between the neutron scattering spectra and the numerical study is reasonable and allows a detailed description for each sample.

1. Introduction

The microemulsions are a thermodynamically stable dispersion in water of oil nanodroplets surrounded by a surfactant film [1]. Microemulsions are widely used in a large array of chemical and biochemical systems including detergents, lubricant, cosmetics, formulation of paints and advanced petrol extraction [2-6]. In recent decades, many studies have focused on mixtures between polymers and surfactant-water-oil systems [7-14]. These systems are of great practical importance, for example in the control of rheology and colloidal stability. Indeed, they serve as a model system for a general class of transient networks. The behavior of these mixed systems depends strongly on the interaction between the polymer and the surfactant film. Generally, for a given number of polymer chains per colloid, they have a monophasic region populated with flower-like colloids with a low droplet concentration, followed by a two-phase region due to attractive phase separation and finally a concentration phase region of connected colloids, with a gel-like behavior [15, 16]. The first theoretical approach was proposed by Milner and Witten, who adapted self-consistent field calculations to telechelic polymer brushes between flat plates [17], Meng and Russel [18], redid this calculation and they obtained a stronger attraction between the plates in better agreement with the experimental results [19, 20]. As regards the curved surfaces, the problem is more complicated, Semenov et al. have proposed a scaling approach [21]. The results for potentials published on telechelic chains connecting finite curvature surfaces come from Bhatia and Russel [22]. They obtained analytical results for plane plates and numerical results for chains between two spheres. Porte and al [23] used a model derived from Bhatia and Russel to analyze experimental spectra of small-angle neutron scattering of microemulsion nanodroplets connected by the telechelic polymers.

In this study, we plan to describe and model interactions between spherical oil-in-water neutral microemulsions nanodroplets with telechelic polymers (PEO-2m), to which small amounts of cationic surfactant Cp⁺ are added. Then, we use a microemulsion of decane stabilized in distilled water by a monolayer of non-ionic surfactant TX100 and co-surfactant TX35. To this microemulsion, a telechelic polymer consisting of a water-soluble poly (ethylene oxide) chain, PEO (abbreviated), to which two hydrophobic ends C12H25 are grafted, it will be noted PEO-2m. Thus, the polymeric ends (stickers) are incorporated into the droplets to bridge them and form a multi-connected network. The presence of Cp⁺
introduces a coulomb repulsive interaction. Our objective is to "offset" the unknown attractive interaction introduced by the addition of PEO-2m, by a known repulsion interaction. We want to model these interactions by an appropriate potential and to find the parameters of good agreement between the SANS experiments and the numerical study for all studied cases.

2. Experimental section

2.1. Materials and Preparation of the microemulsion droplets with polymers PEO-2m

Oil-in-Water microemulsion studied system contain a neutral surfactant TX100, cosurfactant TX35, oil of decane, and Cetylpyridinium Chloride (CpCl). All components are purchased from Fluca (cf. table 1 for details). Samples are prepared in bi-distilled water. The ratio in weight of TX35 to TX100 is set equal to 0.48. The ratio in weight of Decane to surfactant film is 0.76.

To the microemulsion, we add the hydrophobically modified poly (ethylene oxide) PEO-2m. PEO has been hydrophobically modified and purified in the laboratory using the method described in Refs. [24] and [25]. PEO-2m contains an isocyanate group between the alkyl chain C12H25 and the ethylene oxide chain. The samples are characterized by a volume fraction of droplets $\Phi$, the number $r$ of C12 chains per droplets.

To obtain weakly charged nanodroplets, we substitute a small fraction of the non-ionic surfactant TX35 and TX100 with cationic surfactant. The charge rate $\tau$ is the ratio in weight of the cationic surfactant CpCl to the neutral surfactant film. The details on the components are indicated in Table 1.

| Components                                                                 | Molar mass (Dalton) | Density (g/cm$^3$) |
|----------------------------------------------------------------------------|---------------------|--------------------|
| H$_2$O                                                                     | 18                  | 1                  |
| (H$_3$C)$_3$C(CH$_2$)$\_{12}$C$_8$H$_{25}$ (H3C)$_3$C(CH$_2$)$\_{12}$OH (TX100) | 624                 | 1.07               |
| (H$_3$C)$_3$C(CH$_2$)$\_{12}$C$_8$H$_{25}$ (H3C)$_3$C(CH$_2$)$\_{12}$OH (TX35) | 338                 | 1.02               |
| [H$_3$C(CH$_2$)$_{12}$]Cl (Decane)                                          | 142                 | 0.73               |
| [H$_3$C(CH$_2$)$_{15}$][Cl (CpCl)                                           | 339.5               | 1.656              |
| [CH$_3$(CH$_2$)$_{11}$NHCO(OH(2CH$_2$)$_{22}$O(CO)(CH$_2$)$_{11}$CH$_3$ (PEO-2m) | 10400               | 1.2               |

2.2. Small-angle Neutron scattering (SANS) measurements

SANS experiments are carried out on the PACE spectrometer at the LLB-Saclay (France). We obtain intensities in absolute units (cm$^{-1}$) with accuracy better than 10%. To simulate correctly the experimental spectra, all the models spectra are convoluted by the instrumental response function taking into account the uncertainty of the neutron wavelength and angular definition [26]. The scattered SANS intensity $I(q)$ by a dispersion of spherical colloids with a moderately spread distribution of size was modeled using the following expression [27]:

$$I(q) = \frac{1}{V} \left\langle A(q) \cdot A(q) \right\rangle = \Phi \cdot v \cdot (\Delta \rho)^2 \cdot P(q) \cdot S(q)$$

(1)

where $q$(Å$^{-1}$) is the scattering vector, $\Phi$ is the volume fraction of the aggregates, $v$(cm$^3$) is the dry volume of the aggregates, $\Delta \rho=6.6 \times 10^{6}$cm$^{-2}$ is the contrast, i.e., the difference in the scattering length density of the aggregates and of the solvent. $P(q)$ is the form factor of the colloidal aggregates. The analysis of measurable structure factor $S(q)$ allows to extract information on about the interdroplets potential of the system; i.e., the interactions between nanodroplets are repulsive or attractive.

3. Theoretical background

3.1. Expression of the pair potential

The total interaction potential between droplets with PEO-2m and the surfactant Cp$^+$ is written:

$$U_{\text{tot}} (r) = U_{\text{HIS}} (r) + U_{\text{VDW}} (r) + W (r) + V (r) + U_{\text{Coulomb}}$$

(2)

- The simplest potential of pair which is between hard spheres of diameter 2R is:
Here, ‘r’ is the center-to-center distance; R is the radius of the nanodroplets.

- The attractive interaction of van der Waals acts at distances between nanodroplets of the order of 10% of their diameter. In the case of two dispersed spheres, $U_{vdw}$ potential [28]:

$$U_{vdw}(r) = -\frac{A_H}{6} \left( \frac{2R^2}{r^2} \ln \left( \frac{r^2-4R^2}{r^2} \right) + \frac{2R^2}{r^2} \right)$$, for: $r > 2R$

$A_H$ is the effective Hamaker constant, here, we consider $A_H = 1.1 k_B T$, this value is appropriate for decane nanodroplets interacting through water [29]. To the neutral microemulsion, we add telechelic polymers with two end (PEO-2m), an additional attractive and a steric repulsive contribution must be taken into account:

$$V(r) = V_{\text{steric}}\left(\frac{2R}{r}\right) \exp\left(-\frac{r-2R}{\lambda_{\text{steric}}}\right)$$ and $$W(r) = W_{\text{bridging}}\left(\frac{2R}{r}\right) \exp\left(-\frac{r-2R}{\lambda_{\text{bridging}}}\right)$$

where $V_{\text{steric}}$ and $W_{\text{bridging}}$ are the contact potential for the steric and the bridging interaction. The range of interactions of the polymers is set by their size. The parameters have been fixed to an identical interaction range $\lambda_{\text{steric}}$ of 35Å and $\lambda_{\text{bridging}}$ of 100Å.

- When the cationic surfactant $\text{Cp}^+$ is added to the system (microemulsion with PEO-2m), a screened Coulombic repulsive potential [30, 31] is added to the existing interaction:

$$U_{\text{Coulomb}}(r) = k_B T \frac{Z_{\text{eff}} l_B \left( \frac{e^{(r-2R)\beta_{\text{steric}}}}{(1+kR)^2} \right)}{r}$$

The range of repulsive interactions is determined by the Debye length $k^{-1}$, with a Bjerrum length of $l_B = \frac{\varepsilon e^2}{4\pi \varepsilon_0 k_B T} = 7.18 \text{Å}$ and $T = 298 \text{K}$. $Z_{\text{eff}}$ is the number of charge effective per nanodroplets [32].

In the calculation of $I(q)$, $Z_{\text{eff}}$ is the only adjustable parameter.

The total dimensionless pair potential, $\frac{U(r)}{k_B T}$ of expression (2) is:

$$\frac{U(r)}{k_B T} = \begin{cases} \infty & \text{for: } r \leq 2R \\ \frac{1.1}{6} \left( \frac{2R^2}{r^2} \ln \left( \frac{r^2-4R^2}{r^2} \right) + \frac{2R^2}{r^2} \right) + \frac{Z_{\text{eff}} l_B \text{e}^{(r-2R)\beta_{\text{steric}}}}{(1+kR)^2} r & \text{for: } r > 2R \\ \frac{2R \cdot V_{\text{steric}}}{k_B T} \frac{e^{(r-2R)\beta_{\text{steric}}}}{r} + \frac{2R \cdot W_{\text{bridging}}}{k_B T} \frac{e^{(r-2R)\beta_{\text{bridging}}}}{r} \end{cases}$$

### 3.2 Integral Equation Method

The Ornstein–Zernike (OZ) integral equation is written:

$$h(\tilde{r}) = c(\tilde{r}) + \rho \int c(\tilde{r}-\tilde{r'}) h(\tilde{r'}) d\tilde{r}$$

The (OZ) equation related total; $h(\tilde{r}) = g(\tilde{r}) - 1$ and direct $c(\tilde{r})$ correlation functions of two particles at distance ‘r’ apart [33, 34]. To compute $g(r)$, the (OZ) equation was solved using the hypernetted-chain approximation (HNC) [35].

In terms of $c(r)$, the (HNC) closure relation is:

$$c(r) \approx -\beta U(r) + h(r) - \ln[1+h(r)]$$
Exponentiation gives:
\[ g(r) \approx \exp\left[-\beta U(r) + h(r) - c(r)\right] \]  (10)

For an isotropic colloidal fluid, the information on the radially averaged near-range ordering, i.e. the conditional probability of finding the center of colloid nanodroplets at separation \( 'r' \) from another one [36] is encoded in the equilibrium RDF \( g(r) \). In the reciprocal wavenumber space, the same structural information is included in the structure factor \( S(q) \).

4. Results and discussion

4.1 Phase behavior
The phase behavior of the quaternary system (TX_{100}, TX_{35}, decane, water) has been detailed in the literature [37-39]. For a small amount of PEO-2m (\( r < 12m \)), the solution is mono-phasic separated by a percolation line between a sol phase and a gel phase [40]. For the same volume fraction (\( \Phi < 7\% \)) and by increasing the amount of PEO-2m (\( r > 12m \)), one observes an associative phase separation between a concentrated and very diluted a solution [41]. In this study, we investigate samples with \( \Phi = 2.8\% \) and \( r = 29.6-2m \). Thus, in this studied composition, the sample undergoes phase separation and is biphasic. The addition of a POE-2m polymer \( (r = 29.6) \) on a microemulsion will cause an associative phase separation due to the effective attractive interaction between droplets in the low volume fraction. To avoid this phase separation, an adequate amount of cationic surfactant CpCl must be added to the solution. The latter introduces a repulsive interaction (electrostatic effect) which will be compensated by the attractive interaction due to the bridging. Thus, a single-phase solution is obtained. Our particular interest is to "weight" this unknown attractive interaction by a known repulsive interaction.

4.2 Nanodroplets shape and size
From SANS experiments, many studies have been carried out on different systems (bare microemulsion, microemulsion with POE-2m and / or the cationic surfactant (Cp⁺) ) and have shown that the spherical shape and the size of the microemulsion droplets remain identical from \( \Phi = 1.4 \) to 26.5\%, with an average radius \( R = 82\AA \) and a polydispersity \( \Delta R = 15\AA \) [12,23,43].

4.3 Structure Factor of mixture system (microemulsion + PEO-2m + Cp⁺)
In Figures. 1, 2 and 3, the SANS pattern of the bare microemulsion \( (r = 0) \) is compared to that of a microemulsion with PEO-2m \( (r = 29.6-2m) \) at different charges rates \( \tau \) (+0.22\%, +0.5\%, +0.75\% and +1\%).

![Figure 1. Scattered intensity I (q) from SANS and HNC at \( \Phi = 2.8\% \), for bare microemulsion (○ \( r = 0 \)) and with PEO-2m \( (r = 29.6) \), at \( \tau = +0.22\% \) (○)](image1)

![Figure 2. Scattered intensity I (q) from SANS and HNC at \( \Phi = 2.8\% \), for bare microemulsion (○ \( r = 0 \)) and with PEO-2m \( (r = 29.6) \), at \( \tau = +0.22\% \) (○) and \( \tau = +0.5\% \) (○)](image2)
For a low charge rate $\tau = +0.22\%$ (Figure. 1), a significant rise in the scattered intensity is observed for $q \rightarrow 0$ (the osmotic compressibility) and the presence of a correlation bump of a particular distance between the droplets is favored ($d \sim 332\text{Å}$). This distance is imposed by the average end-to-end distance of the telechelic polymers [13]. Furthermore, it's worth noting that the significant increase in intensity is reasonable because the repulsive effect introduced by the charges ($Cp^+$) is very weak compared to the still dominant attractive effect brought by PEO-2m. The best fit of the curve of the sample, using the model described by eq 2 yields an average number of charges per droplet $\bar{p} = 8 (Z_{\text{eff}} = 4.8)$ and a bridging potential $W_{\text{bridging}} = -4.02k_B T$.

Figure 2 displays the variation of the scattered intensity with $q$ for charges rates $\tau = 0, +0.22\%$ and $+0.5\%$. The scattered intensity decreases at low $q$ when the amount of added charges is increased from $\tau = +0.22\%$ to $+0.5\%$, demonstrating that the repulsive effect is reinforced. This is illustrated by the decrease of $I(0)$ from $43000 \text{ cm}^{-1}$ to $16000 \text{ cm}^{-1}$. The shoulder gives a particular distance between the droplets ($d \sim 353\text{Å}$).

Furthermore, an excellent quantitative agreement is found between the experimental and calculated spectra with average number of charges per droplet $\bar{p} = 19.15 (Z_{\text{eff}} = 8)$ and a bridging potential $W_{\text{bridging}} = -3.9k_B T$.

For higher charges rates, $\tau = +0.75\%$ and $+1\%$ (Figure. 3), we note the appearance of a very well defined sharp correlation peak for these two samples. In addition, the maximum of the correlation peak clearly shifts to large $q$’s as $\tau$ increases. Then, the distance between the droplets decreases and the solution becomes more structured.

For $\tau = +0.75\%$, the best fit is obtained with an average number of charges per droplet $\bar{p} = 28.6 (Z_{\text{eff}} = 17)$ and a bridging potential $W_{\text{bridging}} = -2.8k_B T$. For $\tau = +1\%$, $\bar{p} = 38 (Z_{\text{eff}} = 21)$, $W_{\text{bridging}} = -2.5k_B T$.

Regarding the scattered intensity at low $q$, it is observed to decrease when the charge rate $\tau$ increases from $\tau = +0.75\%$ to $\tau = +1\%$. This is a signature of an increasing correlation between the nanodroplets and consequently the repulsive interaction is more reinforced. This evolution shows the attractive interaction introduced by POE-2m is gradually compensated and replaced by Coulomb repulsion. As a result, we can probe the balance of telechelic polymers bridging and Screened-Coulomb Interactions for such systems. Alternatively, we can use a known repulsive interaction (Coulomb interaction) to “weight” a not well-defined attractive interaction (attraction by bridging).

Figure 3. Scattered intensity $I(q)$ from SANS and HNC at $\Phi = 2.8\%$, for bare microemulsion ($\circ \; \tau = 0$) and with PEO-2m ($\circ \; \tau = 29.6$), at $\tau = +0.75\%$ ($\circ$) and $\tau = +1\%$ ($\bullet$).
We now examine the evolution of these samples from their phase behavior when the charge rate $\tau$ is increased. For low charge rates (+0.22%, 0.5%), the attractive effect is always present and it is reminiscent of the existence of some bridges between the droplets. For the large values of $\tau$ (+0.75%, +1%), the attractive effect no longer exists and the solution is completely fluid. This means that the nanodroplets are not connected between them and are instead decorated by loops (the two hydrophobic ends of the polymers stick in the same nanodroplet). As a result, there is a gradual transformation of the bridges connecting the neighbouring nanodroplets into loops. In Table 2, we report all results found for samples studied.

**Table 2.** Comparison of the results for $\Phi = 2.8\%$ of the distance between droplets ($d$), their effective charges $Z_{\text{eff}}$, $I(0)$, the repulsive potential in contact $U_{\text{OC}}$, bridging potential $W_{\text{bridging}}$, and steric potential $V_{\text{steric}}$ for the microemulsion with PEO-2m ($r = 29.6$), at different charge rates of $C_p^+$ $\tau$ (+0.22%, +0.5%, +0.75% and +1%)

| With POE-2m+charges | $I(0)$ (cm$^{-1}$) | $d$ (Å) | $Z_{\text{eff}}$ | $U_{\text{OC}}$ (K$B_T$) | $V_{\text{steric}}$ (K$B_T$) | $W_{\text{bridging}}$ (K$B_T$) |
|----------------------|------------------|--------|-----------------|--------------------------|--------------------------|--------------------------|
| $\tau = +0.22\%$     | 29436            | 332    | 4.8             | 0.37                     | 10.5                     | -4.02                    |
| $\tau = +0.5\%$      | 16640            | 353    | 8               | 1.05                     | 10.5                     | -3.9                     |
| $\tau = +0.75\%$     | 7938             | 409    | 17              | 4.75                     | 10.5                     | -2.8                     |
| $\tau = +1\%$        | 4733             | 487    | 21              | 7.25                     | 10.5                     | -2.5                     |

5. Conclusion

In this paper, we use small angle neutron scattering to “weight” an unknown effective attraction against using a well-known electrostatic repulsion. The scattered intensity can be reproduced qualitatively using the (OZ) equation, with the (HNC) approximation. The interactions induced by the presence of PEO-2m and $C_p^+$ are modeled by Yukawa type potentials. The quantification of the attractive interaction is made by determining the bridging potential in contact $W_{\text{bridging}}$. In each case, we found the effective charge per droplet $Z_{\text{eff}}$ and the Coulombic potential at contact $U_{\text{OC}}$. The strongly increase of the electrostatic repulsion strongly depends on the number of charges per droplet and for the large values of $\tau$, the system is fluid. We obtained a reasonable adjustments for all cases studied. Our results indicate good matching of the parameters of this potential model.

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6. References

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