EPACNUS[28] for lower/middle IFT form straight lines with slope of 1.3, implying that carbon number is a field variable for this interface.

A number of details of other work on this system can be found in the Ph.D. dissertation of Kim.[15]

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Mobility Measurements in Microemulsion Gels

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Gelatin can be solubilized in the water pool of water-in-oil microemulsions formed by AOT (bis(2-ethylhexyl) sodium sulfosuccinate) in isooctane and, under certain conditions, the whole system can be transformed in a transparent, stable gel. A partial physical chemical characterization of these gels is presented, based mostly on viscosity (energy dissipation) and NMR relaxation time measurements, with the aim of investigating the mobility and, more generally, the structure of these novel materials. The viscosity is very high, typical values ranging around 1000 P. However, when the molecular mobility is investigated by pulsed NMR spectroscopy, it is found that isooctane is characterized by T_1 ca. 1.695 s, a value which is very close to the value of the neat solvent. As far as the surfactant is concerned, NMR spectra are characterized by a very high resolution with relaxation times close to those found for AOT in liquid reverse micelles. ^{13}C resonances of AOT suggest a picture of the surfactant in the gel phase, according to which the two ester moieties of the molecules assume a closed form. Gelatin, conversely, is highly rigid and cannot be properly studied by NMR spectroscopy. The chemical shift of the water signal is very similar to data previously published on micelles at very high water content (4.8 ppm); however, the line width in gels is larger by an order of magnitude and increases linearly with an increase in the gelatin content in the water phase. From all of these data taken together, in particular from the apparent contradiction between the high viscosity of the gel and the high mobility of the major component (hydrocarbon), a picture for these gels emerges, according to which very large droplets of organic solvents (400-2000 Å in diameter in the rough assumption of spherical droplets) are entrapped by rigid gelatin networks.

Introduction

In a previous communication, this group has described the preparation of a novel family of gel.[1] The material is prepared from water-in-oil microemulsions; in particular from a solution of AOT (bis(2-ethylhexyl) sodium sulfosuccinate) in a hydrocarbon (e.g., isooctane) containing water, up to a W_0 (W_0 = H$_2$O]/[AOT]) of ca. 30–40. Gelatin is dissolved in the water microphase and, following a simple process of warming up and cooling, the whole organic solution is transformed in a transparent gel. Other groups have also started investigating such systems.[2] The term "microemulsion gels" can be used to define this novel material; more generally, the term "organogels" (or in particular, "hydrocarbon gel") conveys the qualitative information that the largest component (80–90% v/v) is an organic solvent, specifically a hydrocarbon.

Preliminary physicochemical studies have been performed on these gels, but neither their structure nor the mechanism of formation is understood.

In particular, one would like to know how the relatively large amount of organic solvent (ca. 80% v/v of the system) can "gelify" as a result of the presence of a relatively small amount of gelatin which is localized in the remaining water microphase. One would also like to know the distribution of water in the microemulsion gel and establish, for example, whether we are dealing with a

bicontinuous system, or whether water remains localized in droplets as in the starting microemulsion solutions.

These questions are also important in view of the application of the gels. Compartmentation of hydrophilic biomolecules, of enzymes and/or bacteria in the gels, for example (studies on this line are in progress in this group), can be successful only if water is present in the gel material in discrete pools which are large enough to host the guest molecules.

In this paper, the structure of the microemulsion gels has been studied by pulsed NMR spectroscopy. This technique is, in fact, particularly suitable for studying solids whose components have different mobilities.[3] We will show that by investigating the dynamic properties of the various components of the gel (AOT, isooctane, water, and gelatin), a first insight of the overall structure can be gained.

^{1}H and ^{13}C NMR studies of AOT reverse micelles have been already reported in the literature.[4-11] T_1 measurements have been

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performed by Maitra et al.[12] and Martin and Magid.[13]

Experimental Section

Materials. Isooctane (puriss. p.A.) and gelatin Bloom No. 250 were from Fluka. AOT and gelatin Bloom No. 300 were purchased from Sigma and were used without further purification. Bloom number is an indication of the rigidity of gels produced in water, and it is roughly proportional to the molecular weight of the polymer.

Deuteriated benzene and deuteriated water were purchased from Carlo Erba Farmitalia and Merck Sharp & Dohme Canada Ltd (Montreal).

Sample Preparation. Gels were prepared as previously described.[1] The gelatin concentration was used overall (% G_{ov}; w/v) and water pool concentration was % G_{wp} (w/v). The water to surfactant molar ratio is defined by W_0 (W_0 = [H_2O]/[AOT]).

The following samples were investigated (A) Fluka gelatin with Bloom No. 250: (1) G_{ov} 9.5% = [AOT] = 150 mM, W_0 = 40; (2) G_{ov} = 10% [AOT] = 150 mM, W_0 = 60; (3) G_{ov} = 5%, [AOT] = 100 mM, W_0 = 60; (B) Sigma gelatin with Bloom No. 300: (4) G_{ov} = 5%, [AOT] = 150 mM, W_0 = 40; (5) G_{ov} = 6%, [AOT] = 150 mM, W_0 = 60.

Samples were also prepared with deuteriated water under the same conditions.

Methods. Differential scanning calorimetry (DSC) was carried out as described previously.[1] Viscosity and energy dissipation measurements were performed with a Rheometrics dynamic spectrometer (RDS-7700). For this, the sample gel was pressed between the two cylinders (2.5-cm cylinders were used) of the instrument into small films (ca. 0.0598 cm thickness). In order to avoid evaporation of isooctane from the gel, the terminals of the two cylinders were covered with cotton saturated by isooctane before starting the measurements. Indeed, without this precaution, the measured viscosity values tended to increase with time; however, even in this case, the initial viscosity values could be reproduced once isooctance was added with a syringe at the end of the measurements.

For NMR measurements, the following procedure was followed: the gel was first melted at 40–50 °C and the melt was rapidly introduced into small glass tubes (8 mm diameter); the height of the sample in the tube was always less than 1.2 cm in order to have the whole sample within the NMR coil.[14]

For high-resolution ^{1}H and ^{13}C spectra, the same tubes were placed inside a 10-cm standard NMR tube; C_6D_6 was placed in the interspace as an external lock.

Spin–lattice relaxations were measured by the usual inversion recovery technique.

On low-resolution ^{1}H NMR spectra, diode detection, at least 64 different experiments were performed, while for high-resolution ^{13}C NMR spin–lattice measurements, the number of useful points was about 16.

Different delays and numbers of scans, suitable to measure signals very strong or very weak and relaxations spanning between 0.1 and 55 s, were used. In any case, the repetition rate was larger than $5T_1$.

Low-resolution spin–spin relaxation measurements were taken by the usual CPMG technique, observing 512 echoes. All low-resolution NMR spectra were taken at 30 MHz, 20 °C, on a commercial spectrometer "Spinmaster" from Stelar-Mede (PV), Italy.

All high-resolution NMR spectra were taken at 200 MHz for protons and 50.28 MHz for ^{13}C on a Bruker WP200.

Since samples were not degassed, the T_1 and T_2 values reported here cannot be taken as absolute values.

TABLE I: Differential Scanning Calorimetry of Micellar AOT/Isooctane/Water/Gelatin Gels

[AOT]	W_0	% $G(ov)^a$	% $G(wp)^b$	$T_m,^c$ °C	J/g^d
100	40	7	50.7	34.3	31.5
150	40	11	50.5	37.3	32
150	40	10	48	37.1	26
150	30	10.5	56.5	38.3	26.6
150	30	11	57.6	38.3	25

^{a} Overall gelatin concentration (w/v). ^{b} Water pool gelatin concentration (w/v). ^{c} The onset temperature was chosen as melting temperature T_m. ^{d} The value is given in joules/gram of polymer.
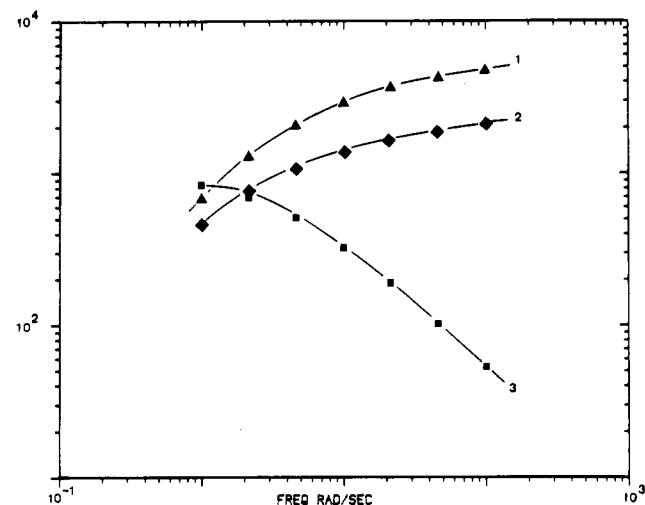

Figure 1. Components of the shear modulus G^* in the AOT/isooctane/water/gelatin gel at 25 °C (gel No. 1 in the Experimental Section). The shear viscosity η^* (curve 3 [poise]), the elastic component G' (curve 1 [dyn·cm^{-2}]) and the viscous component G'' (curve 2 [dyn·cm^{-2}]) are shown versus the frequency [rad·s^{-1}].

Results and Discussion

It is useful to provide first a physical characterization of the gel materials which are under study in the present paper.

Differential scanning calorimetry (DSC) measurements show, in keeping with our earlier findings,[1] that the material prepared in the W_0 range 30–40 and for a gelatin concentration between 7 and 11% (overall concentration w/v) presents a transition in the region 34–38 °C. This is close to the transition observed for water–gel gelatin[15,16] (see Table I).

Viscosity measurements were performed in order to characterize the viscoelastic properties of the organogel.

A typical behavior of the frequency dependence of the elastic component G' of the viscous component G'' and of the shear viscosity η^* is shown in Figure 1. Analogous studies have been carried out (in a smaller frequency region) by Nijenhuis[17,18] for aqueous gelatin gels. In the present work, the storage modulus G' is studied in particular. In Nijenhuis' work, the log G'/log ω behavior is linear in the studied frequency region (−0.5 ≤ log ω ≤ 1.7 rad·s^{-1}), indicating a rubberlike behavior.

In our case a more complex situation is observed. It must be said, however, that, in our case, measurements are made difficult by solvent evaporation (see Experimental Section). In the frequency region 1–100 rad·s^{-1} at large aging times (conditions under which a comparison between our and Nijenhuis' data is possible), the storage modulus G' assumes similar values in the two cases. A comparison of the loss modulus G'' is more difficult, because of the different conditions (aging, concentration); however, it appears that the hydrocarbon gelatin gels have higher G'' values than aqueous gelatin gels (cf. Figure 13 in ref 17 with our Figure 1). Nijenhuis studied in detail the dependence of the storage

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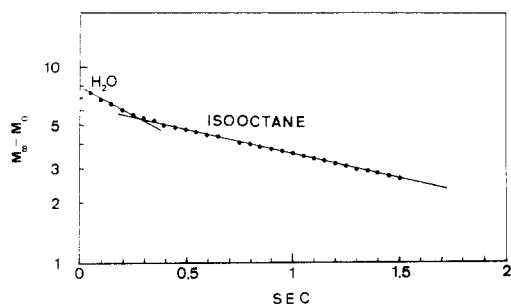
Figure 2. 30-MHz ¹H NMR. Inversion recovery experiment on sample 1a. The double-exponential decay due to the water component is easily observable. For the sake of clarity, only one of every five measured points has been shown in this figure.

TABLE II: 30-MHz Pulsed NMR and T_1 Values for Isooctane and Water Components in Gel

	T_1, s		
sample[a]	isooctane		H_2O
1a	1.696		0.835
1b	1.704		0.878
1c	1.704	1.695 ± 0.010	0.870	0.830 ± 0.050
1d	1.696		0.800
1e	1.678		0.765
2	1.687		1.000
3	1.600		0.900
4	1.704		0.965
5	1.704		1.087
1'[b]	1.670		0.913
neat solv	1.720		

[a] See Experimental Section for the sample description. [b] Same sample as 1 but with deuteriated water.

modulus G' (and partially G'') with aging, which has not yet been performed in our case. Note in Figure 1 that the value of viscosity η^* at low frequency is around 1000 P, which is on the order of the value assumed, for example, for glycerin at −20 °C.

Keeping in mind this very high viscosity of the microemulsion gels, let us consider now their molecular mobility on the basis of NMR experiments.

We have carried out first an analysis at very low resolution (30 MHz) in order to monitor the overall proton relaxation behavior.

Spin–lattice relaxation data of undegassed gel samples are shown in Figure 2. This figure reveals the presence of two components having values $T_1 = 1.695$ s for the major component and $T_1 = 0.835$ s for the minor component, respectively (see Table II). The former can be easily attributed to isooctane, whose relaxation value as neat solvent is about the same, while the latter can be attributed to H_2O, since it is much weaker in gels prepared with D_2O. The corresponding spin–spin relaxation values T_2 are about 70% of the T_1 values, which is normal for undegassed liquid samples.

The observation that the isooctane T_1 values were almost the same in gels as in the neat solvent made it necessary to repeat these measurements many times to make sure that the difference was beyond experimental errors. As shown in Table II, the difference, albeit small, is significant. The observation that the mobility of isooctane in gels is comparable to that of the pure solvent, although surprising in view of the high viscosity of the microemulsion gels, is in agreement with published data on aqueous gelatin gels. In fact, for such samples, Maquet et al.[19] have found that the mobility of water is very high and actually most of the water can be described as bulk water.

The observation of the high mobility of protons in the hydrocarbon gels prompted us to further investigations by means of conventional high-resolution NMR spectroscopy.

In Figure 3, a ¹³C NMR spectrum of a gel is reported. Note the high resolution, which is actually more characteristic of a
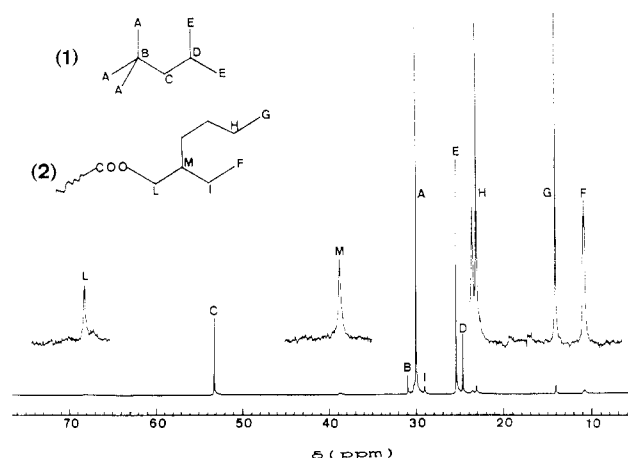

Figure 3. 50.28-MHz ¹³C NMR high-resolution spectrum of sample 1a with peaks assignment.

solution than of a solidlike material. Here it is easy to observe not only the ABCDE peaks due to the isooctane but also some other resonances, weaker and broader, which can be attributed to all the AOT present.

¹³C spin–lattice relaxation values for all isooctane peaks are shown in Table III for several different gels. In order to evaluate the experimental uncertainty, all measurements were repeated on gel samples 1a–e, all having the same isooctane/AOT/water/ gelatin ratio.

For this particular composition, the average values and the corresponding experimental uncertainty have been evaluated, as shown in Table III. Conversely, for the other samples reported in the same table, this laborious procedure (about one day per point measurement) has not been performed.

In the same table, the average value of ¹³C spin–lattice relaxation and their standard deviations measured on a sample of neat solvent are given. It can be noted that all values of ¹³C spin–lattice relaxations measured on the ABCDE peaks of isooctane (see Figure 3, structure 1) in gels are slightly lower than the corresponding values in the neat solvent. The difference is almost within the range of experimental uncertainty, but since it is present for all peaks in all samples and in all measurements, it is physically significant.

As mentioned before, a few ¹³C peaks of AOT are also observable. For those which lie in a spectral range not overlapping any isooctane peak, it is possible to get a spin-lattice relaxation value, as shown in Table IV. It is noteworthy to point out that the ¹³C T_1 values of AOT in gels are close to the values measured in various reverse micelle.[20,21] This indicates that, in particular, the terminal methyl group with a long T_1 value moves rather freely.

For the polar head of AOT, the conformation shown in Figure 4 has recently been suggested by De Marco et al.[6] In the gel phase, ¹³C relaxations not only support this structure, but also give further information about it. In fact, the difference in T_1 values between $-CH_3$ (F) and $-CH_3$ (G) (see Figure 3, structure 2, and Table IV) can be interpreted as arising from a dominant close (Figure 4B) shape of the surfactant molecule, as opposed to the open one (Figure 4A) which was also implicitly assumed by Llor et al.[8] (see Figure 5 in the reference).

In fact, in the open form (Figure 4A), the ¹³C spin–lattice relaxation values of the two methyl groups should be very similar, while in the closed form (Figure 4B), the inner methyl groups should give a lower T_1 value due to steric hindrance. This is indeed experimentally observed in all samples.

T_1 values decrease sharply for peaks HIL (Figure 3, structure 2) and Table IV) and the inner −CH− and −CH₂− inside the polar heads give signals too broad to allow T_1 measurement.

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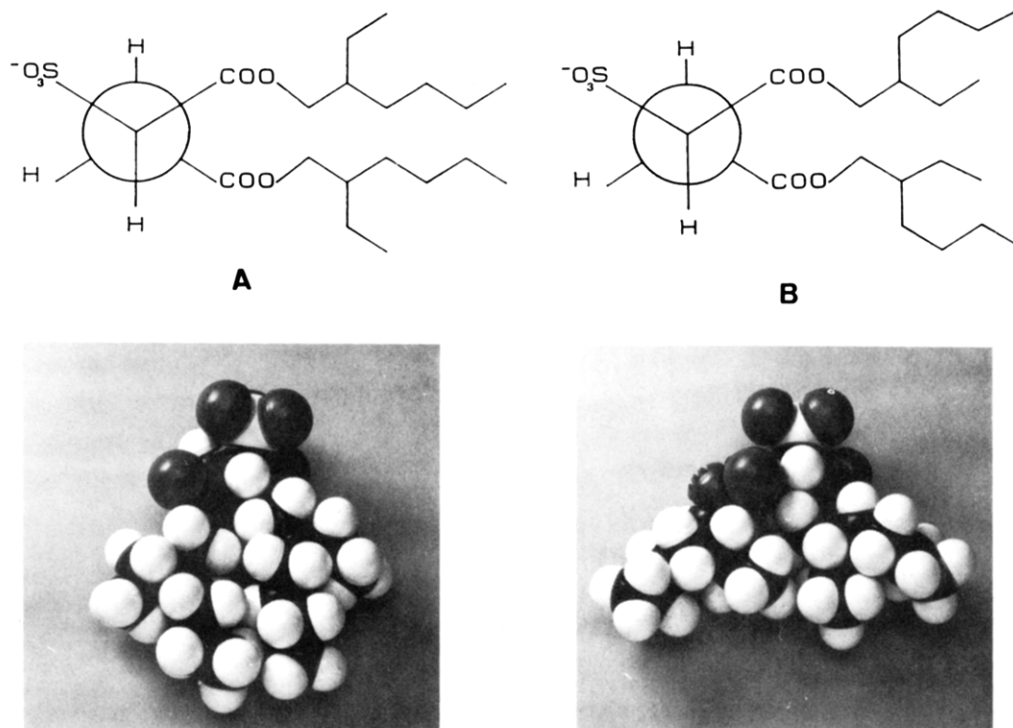
A

B

Figure 4. Drawing and space-filling model of AOT shown using the polar head conformation of AOT proposed by De Marco et al. (see ref 6) for reverse micelles. (A) "Open" and (B) "close" side chain conformation for AOT in gels.

TABLE III: 50.28-MHz ^{13}C High-Resolution NMR T_1 Values for Isooctane in Gels

| | | sample no.^{c} | | | | | | | | | | | |
peak	ppm	1a	1b	1c	1d	1e	1^a	1'^{b}	2'	3'	4	4'	5	5'	neat isooctane
A	29.70	9.1	9.6	8.7	8.7	8.7	8.9 ± 0.4	9.1	10.0	9.3	9.1	9.1	10.0	8.9	10.9
B	30.60							45.2	44.3				55.6	55.6	58.7
C	52.80	13.0	14.3	13.5	13.5	13.5	13.5 ± 0.5	13.0	13.5	14.4	13.9	13.9	14.3	14.5	14.3
D	24.30	20.0	21.7	19.0	19.0	19.1	19.6 ± 1.3	20.0	20.4	18.3	18.7	19.6	21.3	22.2	22.2
E	25.10	10.4	10.0	11.3	10.0	10.0	10.3 ± 0.5	10.4	10.7	10.4	10.8	10.4	11.3	10.9	11.3

^{a} Average value for sample 1. ^{b} The prime indicates that the sample was prepared with deuteriated water. ^{c} See Experimental Section.

TABLE IV: 50.28-MHz ^{13}C High-Resolution NMR T_1 Values for Selected Peaks of AOT in Gels

| | | sample no.^{a} | | | | | | | |
peak	ppm	1	1'^{b}	2'	3'	4	4'	5	5'
F	10.58	1.17	1.10	1.00	1.10	1.26	1.34	1.17	1.13
G	13.88	1.73	1.48	1.56	1.65	1.90	1.70	1.65	1.74
H	22.94		0.70	0.78	0.82	0.69	0.70	0.78	0.61
M	38.60		0.25	0.20	0.21	0.26	0.30	0.23	0.22
L	68.15		0.10	0.10	0.07	0.13	0.10	0.12	

^{a} See Experimental Section. ^{b} The prime indicates that the sample was prepared with deuteriated water.

By assuming that the dominant mechanism of relaxation is the dipolar one, a rough estimate of the correlation times can be made. For the terminal methyl groups, a value of $\tau \approx 10^{-10}$ s is obtained; while for the $-CH_2-O-$ peak, having $T_1 \approx 0.1$ s, the function T_1 vs τ is close to the flat minimum.[22,23] Thus, values estimated from T_1 measurements can be affected by large errors. However, since the nearby CH has a T_1 value about twice as large, the dipolar mechanism of relaxation seems to effectively be the dominant one, and a correlation time of $\approx 10^{-9}$ s can be safely assumed.

This value compares well with correlation times obtained by spin label ESR methods.[24]

Since isooctane shows long T_1 values associated with high mobility, and since T_1 values of AOT show longer values on the hydrophobic end of the molecule and shorter T_1 near the polar

heads, it is reasonable to assume a contact between the hydrophobic part of AOT and isooctane.

All relaxation data, taken together, point to a model for the gel system in which a rigid network of gelatin, water, and the AOT polar head delimitates rather large cavities filled with isooctane. It is possible to roughly estimate, from our data, the average number of isooctane molecules in a cavity. To this aim, let us assume that the relaxation time of the $-CH_2-$ (see C in Figure 3) of isooctane in contact with AOT molecules ("external" isooctane) has the same relaxation time value (0.7 s) as the "external" $-CH_2-$ (see H if Figure 3) of AOT (i.e., isooctane molecules on the surface of the cavity which are in contact with the AOT interface).

Then it is possible to write

$$\frac{1}{T_{gel}} = n\frac{1}{T_A} + m\frac{1}{T_B} \tag{1}$$

where n is the molar fraction of isooctane molecules in the bulk

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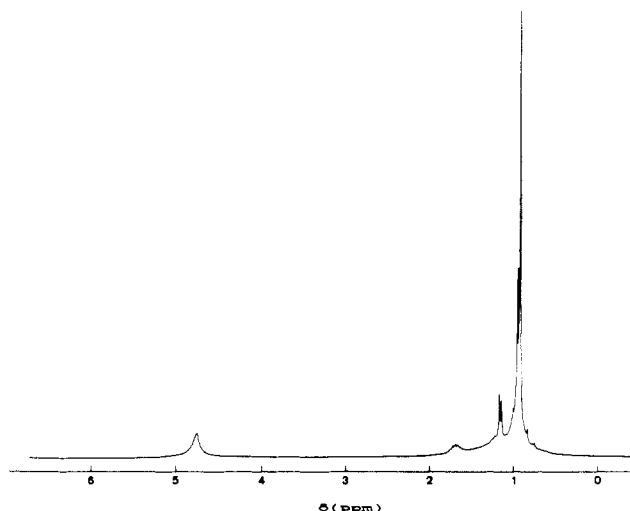(24) Haering, G.; Hauser, H.; Luisi, P. L., manuscript in preparation.

Figure 5. 200-MHz ^{1}H NMR high-resolution spectrum of a isooctane-/AOT gel with the following composition: G_{ov} = 9.5% [AOT] = 150 mM, and W_0 = 40.

of the cavity, T_A the corresponding relaxation time, T_B the relaxation time of the external $-CH_2-$ of AOT (0.7 s), and m the molar fraction of internal isooctane molecules. T_{gel} is the overall relaxation time of the $-CH_2-$ groups in the gel.

Assuming now in first approximation that the cavities are spherical, a simple relation between m and n can be derived

$$m/n = 3/r \qquad (2)$$

where r is the average radius of the sphere. Thus, eq 1 finally relates the experimental values of relaxation time to r. On the basis of a best fitting procedure, it is possible to evaluate an r value which is consistent ($\pm5\%$) with the experimental results.

This calculation yields a diameter of 400–500 Å. Since this is calculated under the rough hypothesis of a spherical cavity, it must be taken as a lower limit. If one considers in fact an irregularly shaped surface, the surface may increase up to 4–5 times, which would mean that cavity diameters up to 2000 Å should be possible (which would agree with preliminary electron microscopy data[1]). This means that isooctane is present in the gel in large droplets, probably connected to one another.

Let us now consider the resonance of water protons.

The chemical shift of the water signal is very similar to data previously published[4-10] on micelles at very high water content (4.8 ppm); however, the line width in gels is larger by a factor of 10 and increases linearly with an increase in the gelatin content in the water phase (see Figures 3 in ref 1).

As mentioned before, T_1 values for water, at 30 MHz, range between 0.8 and 1 s in all gels.

The proton high-resolution spectrum at 200 MHz (Figure 5) clearly shows a peak at ca. 4.8 ppm, with a T_1 value of about 0.8 s. Since this T_1 value is almost independent of the frequency, the correlation time must lie on the left side of the T_1 vs τ function.[25]

No simple dependence of relaxation data on the correlation time can account for both T_1 and the line width. Further work is in progress in order to give an interpretation of the unusual NMR data observed for water.

Since our experiments were performed without degassing the sample, one would expect it to be rather difficult to compare our data with spin–lattice relaxations reported by Llor et al.[8] However, their reported value at 250 MHz at the highest W_0 for H_2O is 0.9–1.0 s, and at 60 MHz, 1.1 s. These values compare surprisingly well with our data (0.8–1.0 s at 30 MHz) in gels.

Relaxation time measurements of gelatin dissolved in D_2O have been also performed. Here, in order to avoid contaminations from water, the gelatin was dissolved and lyophilized from D_2O three times before the final sample preparation. At 30 MHz the relaxation values are $T_1 \approx 50$ ms, T_2 = 3 ms, i.e., very short compared to both isooctane and water relaxations.

This explains why, in the ^{13}C high-resolution spectrum, no peak assignable to the gelatin component is measured (they are too broad to be observed).

Concluding Remarks

The most striking property of the microemulsion gels, arising from this first physicochemical analysis, is the combination of a very high overall viscosity and a very high molecular mobility of the major components. The structure which is compatible with this apparent paradox is a network with a very rigid framework and very large cavities.

Beyond this qualitative picture, however, structural details still remain unknown. In particular, the precise localization and compartmentation of water and AOT must be clarified. Since gelatin is per se not soluble in isooctane, it is most likely that AOT is localized all around the long rigid macromolecules, with water in between. However, the data relative to water do not lend themselves to simple interpretation. There are probably two or more states of water in these microemulsion gels, perhaps with the original micellar structure still partly present.

Investigations of these and novel gels (in which lecithin replaces AOT and gelatin[26]) are continuing with NMR, with the ESR technique, as well as with different microscopy techniques.

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