INVESTIGATION OF SPREAD AND ADSORBED MONOLAYERS OF XANTHAN

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In EOR the biopolymer Xanthan is applied as an additive to injection fluids to enhance mobility control. Xanthan may either be supplied as a broth or dry powder. Dependent on this xanthan exhibit different solution behaviour (Littmann, 1989). Different xanthans have been examined regarding surface activity and possible adsorption at the air/water (A/W) interface in order to understand differences in flow behaviour in porous material.

TABLE 1

Xanthan properties: Pyruvate/acetate Conc. %(w/w). Molecular weight (MW)

Code	Type	C_{pyr} %	C_{ace}	MW 10^6g/mol
A	Broth	2.87	1.87	12.7
B	Broth	3.13	5.34	8.5
C	Broth	4.68	3.39	-
D	Broth	4.54	3.93	-
E	Powder	3.53	4.88	10.5

Both surface tension (γ) and pressure (π) was measured by the Wilhelmy plate method using a Sartorius balance. π was recorded as a function of surface compression using a Langmuir balance set-up. Xanthan was spread using a microsyringe. Xanthan properties are given in Table 1. γ was measured as a function of concentration (C) and temperature (T). For commercial xanthan powder spread monolayers were investigated.

The surface tension of different xanthans was measured as a function of concentration (C) as given in Fig.1. Xanthan broth showed decrease of γ with increasing C whereas the powder products had little effect on γ at ambient temperature. This may be ascribed to the presence of broth residuals, and possible interactions between these and the xanthan molecules. In the powder products the double helix conformation may be dominating in solutions leading to less surface activity in accord with surface conformation of proteins (Birdi, 1990). The surface activity of xanthan broths may explain the need for decreasing the residual oil saturation in numerical reservoir

simulations as reported elsewhere (Littmann & Kleinitz, 1990).

The purified xanthans however became surface active upon heating as indicated by Fig. 2, π vs T. As can be seen the slope of the curve is significantly changed as the temperature exceeds ca.47°C. This may be related to the partial unfolding of the xanthan double helical structure leading to a change in the adsorption equilibrium as the single stranded segments are expected to show enhanced adsorption at the A/W interface.

Monolayers of spread purified xan-than were studied regarding the effects of T and salinity. No rise in surface pressure was observed on compression of xanthan on distilled water. At 2 M NaCl subphases xanthan formed condensed monolayers, showing no rise in π untill highly compressed, Fig.3. The effect of T on π-A isotherms of spread xanthan was seen as an increase in the magnitude of A(inf)(see Fig.3), Fig.4. This is assumed related to the conformational expansion of the xanthan molecule explaining the change in surface activity seen in Fig.2.

The presence of proteins in xanthan solutions have been reported to promote microgel formation (). Hence mixed monolayers of Bovine Serum Albumin and xanthan were studied on various substrates. These measurements indicated that interaction between xanthan and proteins is primary takes place in the bulk phase, whereas the protein seems to exclude xanthan from the interface.

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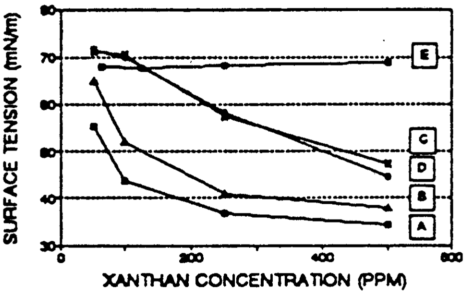

FIG. 1. Surface tension vs xanthan conc.(ppm). Xanthan code in Table 1.

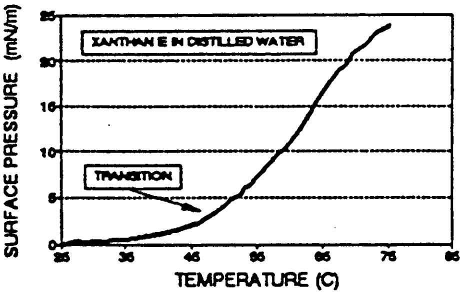

FIG. 2. Surface pressure vs Temperature. Xanthan E in Dist. water.

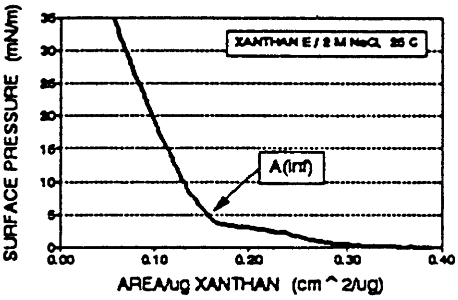

FIG 3. Monolayer π-A isotherm. mN/m vs $cm^2/\mu g$. Xanthan E at 2M NaCl. 25°C.

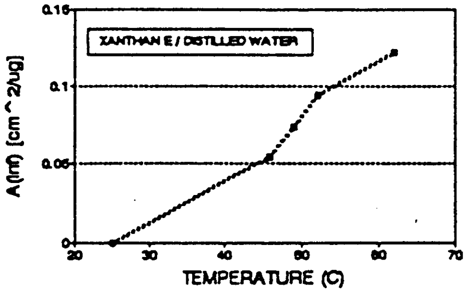

FIG 4. Area at inflection (A(inf)) vs Temperature. Xanthan E at Dist. water.

MONOLAYER (π-A) STUDIES OF XANTHAN POLYMERS

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ABSTRACT.

The biopolymer xanthan may be applied as an additive to injection fluids in improved oil recovery (IOR). The xanthans are supplied either as a fermentation broth or a purified dry xanthan powder. Dependent on this the xanthan exhibit different solution behaviour. Xanthan solution based on xanthan powder exhibit hardly any surface activity at ambient temperature. Spread films of xanthan solutions were investigated using the Langmuir balance. With increasing salinity or temperature an increase in the surface pressure (π) during compression was observed. No rise in surface pressure was seen on compression of xanthan on distilled water at ambient temperature. With increasing ionic strength of the subphase the xanthan monolayer becomes more condensed. At increased temperature a partial unfolding of the helical structure takes place, leading to a change in the adsorption equilibrium as the single stranded segments show increased adsorption. A surface conformation, which explains the change in surface activity upon heating, is proposed. From the π-A isotherms the apparent molecular weight is calculated and the fraction of xanthan at the surface is determined using the molecular weight of the xanthan as measured by Size Exclusion Chromatography. The results supports the conformational model.

INTRODUCTION.

The biopolymer xanthan has a widespread commercial exploitation. In food industry it has been applied as thickener agent, emulsion stabilizer and as gelling agent in combination with locust bean gum. Other industrial uses range from suspending agents for paints to viscosity control in improved oil recovery (IOR) by polymer flooding |1|.

Xanthan is produced as an extracellular polysaccharide by fermentation of Xanthomonas Campestris. The primary structure of the macromolecule consist of a cellulose-like main chain. The chain is substituted in the O-3 position at every second glucose residue with a β-D-Man-(1-4)-β-D-GlcA-(1-2)-α-D-Man trisaccharide. The internal mannose is acetylated in the O-6 position and the terminal mannose can be substituted with a pyruvate group. The content of acetyl and pyruvate may vary dependent on source and treatment of the xanthan. In a number of reports the content of pyruvate and acetate has been correlated with different solution properties such as viscosity and stability |1,3|.

In IOR the use of xanthan is greatly favored by the stability towards saline stress, temperature and mechanical degradation. Conformational properties is however important for the advanced use in polymer flooding in high-salinity, high-temperature reservoirs as physical properties such as viscosity, salt tolarance and injectability are dependent on the xanthan conformation in solution | 4 |.

In solution the xanthans have been shown to obtain a number of different conformations: helical, random coil, aggregates and microgels as have been visualized by Stokke et al. |5|.

Xanthan may either be supplied as a broth or a dry powder, and dependent on this xanthan exhibit different solution behaviour, and in polymer flooding the injectability is highly affected |6|. In the present study xanthans from broth and from commercial available powders has been examined. Additionally three broth xanthans has been purified to investigate differences between broth and powder products.

Different xanthans have been examined regarding the surface activity and possible adsorption at the air/water (A/W) interface in order to understand differences in flow behaviour in porous material. The behaviour of xanthan at the air/water interface has been investigated by the compression of spread monolayers using a modified Langmuir balance.

EXPERIMENTAL.

All surface pressure measurements have been performed using the Wilhelmy plate method. In the Wilhelmy set-up a platinum plate attached to a Sartorius microbalance interfaced with a PC.

Investigations of the surface pressure dependence on temperature (π-T) was performed by increasing the temperature and measuring π simultaneously. The accuracy of the temperature measurement was approximately ± 3K due to the trough geometry.

Monolayer studies were performed using a Langmuir teflon through with two moveable teflon barriers and a Wilhelmy set up.
The initial surface area is 800.5 cm^2, and the final compressed surface area is 113.6 cm^2. The compression speed was 10 cm/min. By applying a rather high rate of compression effects caused by desorption of the soluble macromolecules in the monolayer may be avoided.

Both Xanthan broth and commercial xanthan powders were investigated in order to compare these products. Three xanthan broths were purified regarding the xanthan by iso-propanol precipitation (3:1), filtration on 0.45 μm Milipore filters and rinsing with iso-propanol. Additionally a powder xanthan product, the Glucomannan (Serva), was investigated. The properties of the investigated xanthans, Pyruvate (P%) and Acetate (A%) content, the concentration of xanthan in the sample (Cact%), the apparent molecular weight (MW) and the elemental composition (C,H,N,S,O) are given in table 1 and 2.

The elemental analysis indicates that the powder products contain some impurities as nitrogen and sulfur, which are not present in the xanthan structure. A mass balance indicates that the impurities to some extent is adsorbed water as both the H and O content is higher than the theoretical value.

Xanthan solutions were prepared in distilled water or NaCl solutions and xanthan were dispersed by short (10 min) ultrasonic treatment (low frequency). This procedure was used instead of other methods applied in the literature such as heating to 90°C and longterm stirring. No effect was seen from the ultrasonic treatment.

All solutions were stored below 5°C, and solutions were examined within two days.

RESULTS.

Within the current work both spread monolayers as well as bulk solution surface tension behaviour have been examined.

Bulk Solutions

The surface tension of aqueous solutions of different xanthans (broth or powder) has previously been measured as a function of xanthan concentration. Xanthan broth showed decreasing surface tension with increasing concentration (about 40 mN/m at 500 ppm), whereas the powder products and purified xanthans showed apparently no dependence of concentration, and only a small lowering (1 - 2 mN/m) of the surface tension at ambient temperature as reported recently by the current authors |7|. The surface activity of broth solutions was mainly ascribed to other broth constituents than xanthan.

Effect of temperature and salinity: Although the purified xanthans exhibit no surface activity at room temperature the effect of temperature on the surface tension of xanthan solutions was investigated. Temperature and surface pressure π (= γ_0 - γ) was measured simultaneously as the sample was slowly heated from 25 to ca. 75°C.

These experiments revealed that the purified xanthan becomes surface active upon heating as indicated in Figure 1 , π vs T. As can be seen the slope of the curve changes significantly as the temperature exceeds ca. 47°C for xanthan E in distilled water. This can be assumed to be related to the well established transition from ordered to disordered conformations with increasing temperature |8|. The same shift in π was seen for xanthan G and F with a slight difference in temperatures.

The temperature at which π changes is shifted by changes in salinity, seen as a displacement of the π-T curves towards lower temperatures. The above mentioned order-disorder transition temperature in the bulk is however known to be increased by increasing salinity. According to the literature we would expect that the high salinity leads to an almost complete stabilization of the xanthan in its ordered conformation, and hence a large increase in transition temperature.

These discrepancies in behaviour can only be explained by a significant difference in bulk properties and surface properties. A possible explanation of the effect of external salt is that salinity has more significant impact on the adsorption at the interface than on the order-disorder transition in the bulk.

The negative Gibbs excess of NaCl at the air/water interface may also give rise to a somewhat different adsorption at the surface in saline solutions due to the salt gradient. The rather complex behaviour of the π-T dependence may however not be explained solely by this. Further work is needed at this level.

Spread Monolayers

The adsorption of spread monolayers were investigated using the Langmuir balance. π-A isotherms were recorded for 6 different xanthans. The effect of subphase salinity (NaCl) and temperature was examined. The effect of temperature has previously been reported but the results are summarized below in order to provide evidence for the proposed surface conformation |7|. Three xanthans were commercial products whereas two were purified by isopropanol precipitation from xanthan broths. As xanthan broth contain large quantities of surface active impurities these cannot be investigated regarding the xanthan behaviour by this method, as the additional material gives rise to very expanded isotherms.

Salt effects

As indicated in Figure 2 no rise in surface pressure was seen upon compression of xanthan E on distilled water. As the salinity (NaCl) of the subphase increased from 0 to 2 M NaCl the surface pressure increases upon compression indicating an increased adsorption of xanthan molecules at the air/water interface.

In 2M NaCl the π-A isotherm is somewhat altered relative to the lower salt concentrations. This could be caused by a slightly change in the monolayer composition. This finding indicates that a major force stabilizing the monolayer is the formation of strong hydrogen bonds between the substrate and the polar groups of the molecules in the monolayer |9|.

The similarity in shape of the curves indicate that more and more material is present in the monolayer as the salinity increases. This can be related to a lower desorption if material into the subphase due to the lower solubility of xanthan. The inflection point A_{inf} was found to vary almost linearly with increasing salinity indicating the increased adsorption in the surface region.

Temperature effect

The effect of temperature on the xanthan molecular conformation in solution is known to be a transition from an ordered (helix) to a disordered (coil) conformation in the bulk phase as mentioned above |8|. The recent presented results showed that π-A isotherms of xanthan E indicated an increased expansion of the molecules in the surface film, and an increased adsorption of xanthan at the air/water interface with increasing temperature even in distilled water. The curve of inflection area versus temperature increases almost linear indicating the expansion of the monolayer |7|.

Evaluation of molecular weights from π-A data

In the case of biopolymer monolayers it provides much information if the π-A data are evaluated according to the virial equation

$$\pi A_{tot} = w_p RT/M_p + b_1 \pi + b_2 \pi^2 + \ldots \qquad [1]$$

where A_{tot} is the total surface area, w_p the amount of polymer at the surface, M_p the molecular weight of the polymer, and b_i the virial coefficients.

By truncation the above equation may be given as

$$\pi A_{tot} = w_p RT/M_p + b_1 \pi \qquad [2]$$

which can be rearranged by introducing $A_w = A_{tot}/w_p = A/M_p$:

$$\pi A_w = RT/M_p + (b_1/w_p)\, \pi \qquad [3]$$

Comparing with the non-ideal film equation of state $[\pi(A-A_0) = RT]$, it appears that the second virial coefficient may be identified with the excluded area. Hence we can write Eq. 3 as:

$$\pi A_w = RT/M_p + A_{w0}\, \pi \qquad [4]$$

where A_w and A_{w0} are respectively the surface area and the excluded area based on weight |10|.

From the above given equations the apparent molecular weight M_p was calculated from plots of πA versus π as indicate in figure 3.

From the magnitude of M_p ($M(\pi$-A)) the fraction f of the molecule at the surface was calculated from molecular weight data obtained by size exclusion chromatography M(SEC) |11|. The data is given table 3. Different solution properties have been reported correlated with the structural composition of the xanthan |3|. A linear regression correlation analysis between MW and the parameters P (pyruvate %), A (acetate %) and P/A was performed using single, paired and triplet parameters. The best regression coefficient ($r^2 = 0.91$) was obtained with the triplet MW = f(P, A, P/A) indicating a lowering of MW with increasing P and an increase caused by an increase in A and P/A. The correlation obtained

$$MW_{app}/10^5 = -61.2 - 15.7P + 15.8A + 63.0P/A, \quad r^2 = 0.91$$

gives good estimation of the apparent molecular weight as calculated from π-A isotherms. The ratio of pyruvate to acetate (P/A) was found to be important in the matching of the correlation. This ratio has in the work of Smith et al. | 12 | been reported to be linear correlated with the order-disorder transition temperature.

DISCUSSION

From the above mentioned results it appears that the phenomena taking place at the air-water interface differs significantly from the behavior of xanthan in bulk.

Xanthan was found to become surface active upon at elevated temperature. A biopolymer showing some structural resemblance with xanthan, galactomannan, has been reported to increase oil-in-water emulsion stability not only by means of viscous effects but also due to interfacial activity |13|.

As described the surface activity increases with increasing temperature for both monolayers and bulk surface tension measurements. This may be ascribed to the increasing disorder of the xanthan molecules as the well known transition from a (double) helical to a random coil like conformation takes place 8 . From this point of view we may state that the adsorption of xanthan at the air-water interface seems to be favored by the disordered conformation.

Increasing subphase salinity does however also increase the adsorption of xanthan at the A-W surface as was seen in the π-A isotherms. This may seem somewhat confusing as increasing ionic strength of the media is known to stabilize the ordered conformation of xanthan in the bulk. Since no adsorption at all can be detected when compressing on a salt-free solution (i.e. when xanthan is in a less ordered conformation) it appears that the presence of salt induces an adsorption rather than a desorption as it could be expected from the temperature-conformation relation given above. In the interpretation we should however bear in mind that the xanthan was spread from distilled water solutions. The spreading media significantly affects the isotherm. The largest change in surface pressure was obtained by spreading from distilled water. The xanthan examined was impossible to dissolve in 2 M NaCl and contained significant amounts of un-hydrated material - the so-called "fish-eyes". The spreading pressures π_s and final compression surface pressures π_f are listed in table 3 for three different spreading media. These results indicate that the conformation obtained in the spreading media is somehow "locked" as it is applied to the high salinity substrate. A similar difference in relation between solution behaviour (viscosity) and solution preparation has been reported for carboxymethylcelullose (CMC). When CMC is dissolved in saline solutions the final solution viscosity is far below the viscosity obtained for CMC solutions made saline preceeding CMC dissolution |1|. This and the above findings for xanthan is assumed related to aggregated solution behaviour when the polyelectrolytes are dissolved in saline media. The low π_f of the prehydrated saline solution may be caused by chain entanglement leading to an increased viscosity of the resulting solution.

The salt effect can be related to a decrease in the solubility of xanthan with increasing salinity, hence a decrease in the loss of xanthan to the subphase as can be seen in the case of protein monolayers |14|. The high ionic strength may also lead to a more compact conformation of the molecule in the monolayer, although the above mentioned negative Gibbs excess of NaCl will decrease the local ionic strength in the surface region. Hence it can be concluded that the first mechanism is responsible to a large extent for the formation of the monolayer. Although xanthan is stable in high salinity aqueous media, when prehydrated in distilled water, a certain decrease in solubility is expected due to the more compact conformation.

In addition the determination of the apparent molecular weight from the π-A isotherms revealed that only a minor fraction of the xanthan molecule is present in the surface region, under the assumption of complete adsorption during spreading on salt containing media. The differences observed in M_{app} between the different xanthans may however not be easily explained, as they may result from as well variations in pyruvate/acetate substitution and molecular weight as it may reflect differences in the thermal history of the xanthans.

As indicated by the magnitude of f only a small fraction of the molecules are present in the surface based on this the below given surface conformation has been proposed. Note that although f is as low as 4% for the xanthan E, this corresponds to approximately 450 pentasaccharide repeat units anchoring to the air/water interface for each xanthan molecule.

Xanthan surface conformation.

Based on the above presented result a surface conformation model of xanthan molecules at the air/water interface has been developed.

In bulk solution xanthan may adapt a conformation consisting of partly ordered, partly disordered chain segments. With this in mind we propose a surface conformation model for xanthan as outlined in the figure below. Figure 4. The model assumes the disordered segments to be present at the air/water interface whereas the helical rod-like dimer submerges into the subphase. Whether or not adsorption will occur in a given conformation is mainly a question whether the gain in entropy upon adapting a suitable conformation on the surface can outbalance the enthalpic term arising from breaking of the stabilizing intramolecular bonds and the xanthan-water interactions. From the study of protein monolayers it has been shown that certain proteins may adapt surface conformations in which the highly ordered helical segments submerges into the substrate whereas the amino acid residues in the less ordered segments are displaced in the surface region |10|. Considering that our calculations show that only a small fraction of the xanthan molecule is present in the surface, it was obvious to propose a surface conformation model for xanthan similar to that of proteins.

When only considering the structure of the xanthan molecule a uniform ratio between hydrophilic and hydrophobic sites is expected. The bulk conformation proposed by Liu et al. |15| may however induce a certain difference in hydrophilicity within the xanthan dimer. This difference may for instance be caused by a rather high hydratization of the compact double helical structure, which hence becomes the more hydrophilic compared to the less ordered segments. In addition the acetyl function on the first mannose residue will be shielded from interactions with water in the helical part as the sidechain fold along the main chain. In the disordered segment the acetyl will give rise to hydrophobic interactions with the solvent. This is in accord with the increased surface activity of the investigated xanthan with increasing temperature as the xanthan becomes more disordered. As the order decreases the disorder propagates the end towards the middle of the molecule |16|.

The xanthans present in the rod-like double helical conformation is hence not likely to be stable in a monolayer.

Interchain interactions will mainly take place through hydrogen bonding and Van der Waals forces. At close packing electrostatic repulsion may occur as well, however the high ionic strength may give rise to a significant charge screening.

The model combines the conformational models for dissolved xanthans present in the bulk-phase proposed by Muller and Lecourtier |17| and the "worm-like dimer" model presented by Sato and coworkers |15|.

All the xanthans examined exhibit condensed π-A isotherms which show no rise in π until highly compressed. At low compressions no interactions take place between the bulky submerging double-helical rod-like segment. As the compression increases the interaction between both the random coil segments in the surface and submerging parts increases. The final steep rise in π is believed to be due to strong interactions between the submerging rods.

CONCLUSION.

The surface activity and monolayer compression of xanthans have been examined. From surface pressure (π) measurements of xanthan solutions it was seen that π increases with increasing temperature, and that π increases significantly above a certain temperature apparently due to the unfolding of the molecule.

Monolayer π-A isotherms revealed that condensed monolayers were formed when xanthans were spread from distilled water solutions onto 2 M NaCl. π-A isotherms at increasing temperature showed that more xanthan adsorb to the surface as temperature increases. Base on this a surface conformation model has been proposed. According to this, the disordered segments adsorb to the surface and the double-helical part of the xanthan dimer submerges into the substrate. This explains the increased adsorption at elevated temperature. calculations of apparent molecular weights from the π-A isotherms indicated that only a small fraction of the molecule is present at the interface when compared to size exclusion chromatographic results.

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LEGENDS TO FIGURES

Figure 1: Surface pressure versus temperature of 500 ppm xanthan solution. Xanthan E.

Figure 2: Monolayer surface pressure versus area isotherms for xanthan E spread on subphases of increasing ionic strength at 25°C.

Figure 3: πA versus π, Molecular weight calculation for xanthan E at 25°C. Non-ideal film behaviour.

Figure 4: Xanthan Surface Conformation as proposed from the presented results.

Table 1. Xanthan code, content of pyruvate P% and acetate A%, activity of material, and molecular weight MW by Size Exclusion Chromatography.

Code	Type	P%	A%	C_{act}%	MW (E-6 g/mol)
A	Broth	2.87	1.87	1.43	12.71
B	Broth	1.7	-	-	8.54
C	Broth	4.68	3.39	2.90	-
D	Broth	4.54	3.93	2.70	-
E	Powder	3.53	4.88	96	10.58
F	Powder	1.79	4.31	ca. 95	-
G	Powder	4.12	5.54	ca. 95	-
H	Broth	3.87	5.82	2.79	-

Table 2. Elemental analysis of investigated products. Theoretical calculation on H-form 1 pyr.,1 ace. Prefix p indicates purified from broth product.* Oxygen by difference.

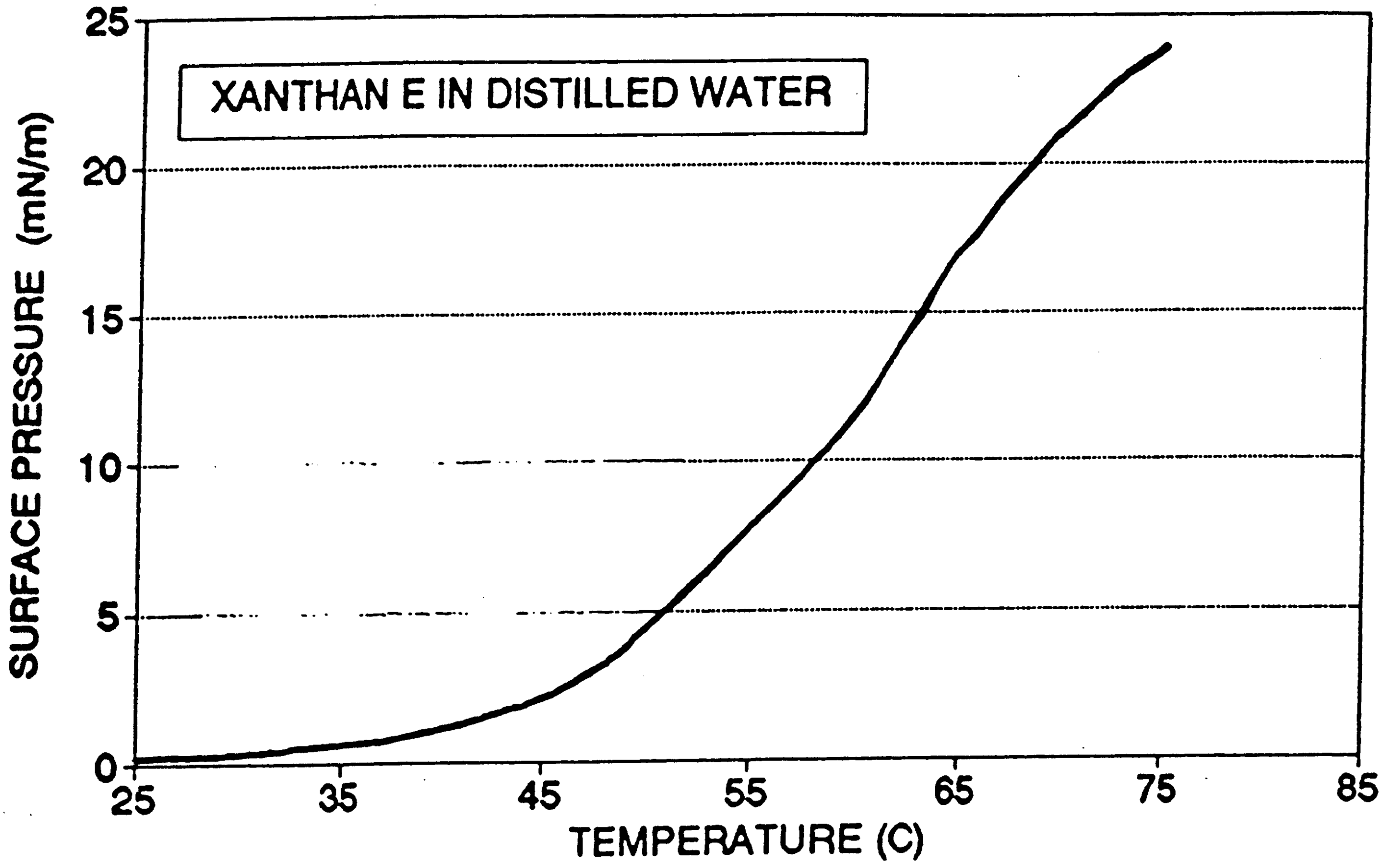
Sample	C	H	N	S	O*
Ap	38.23	6.36	1.98	0.29	53.15
Bp	37.35	6.55	-	0.15	55.96
Dp	34.49	5.88	-	1.41	58.23
E	37.20	6.18	0.88	0.17	55.57
F	38.40	6.25	1.20	0.51	53.64
G	37.38	6.03	0.74	0.21	55.64
Theory	44.9	5.6	0.00	0.00	49.6

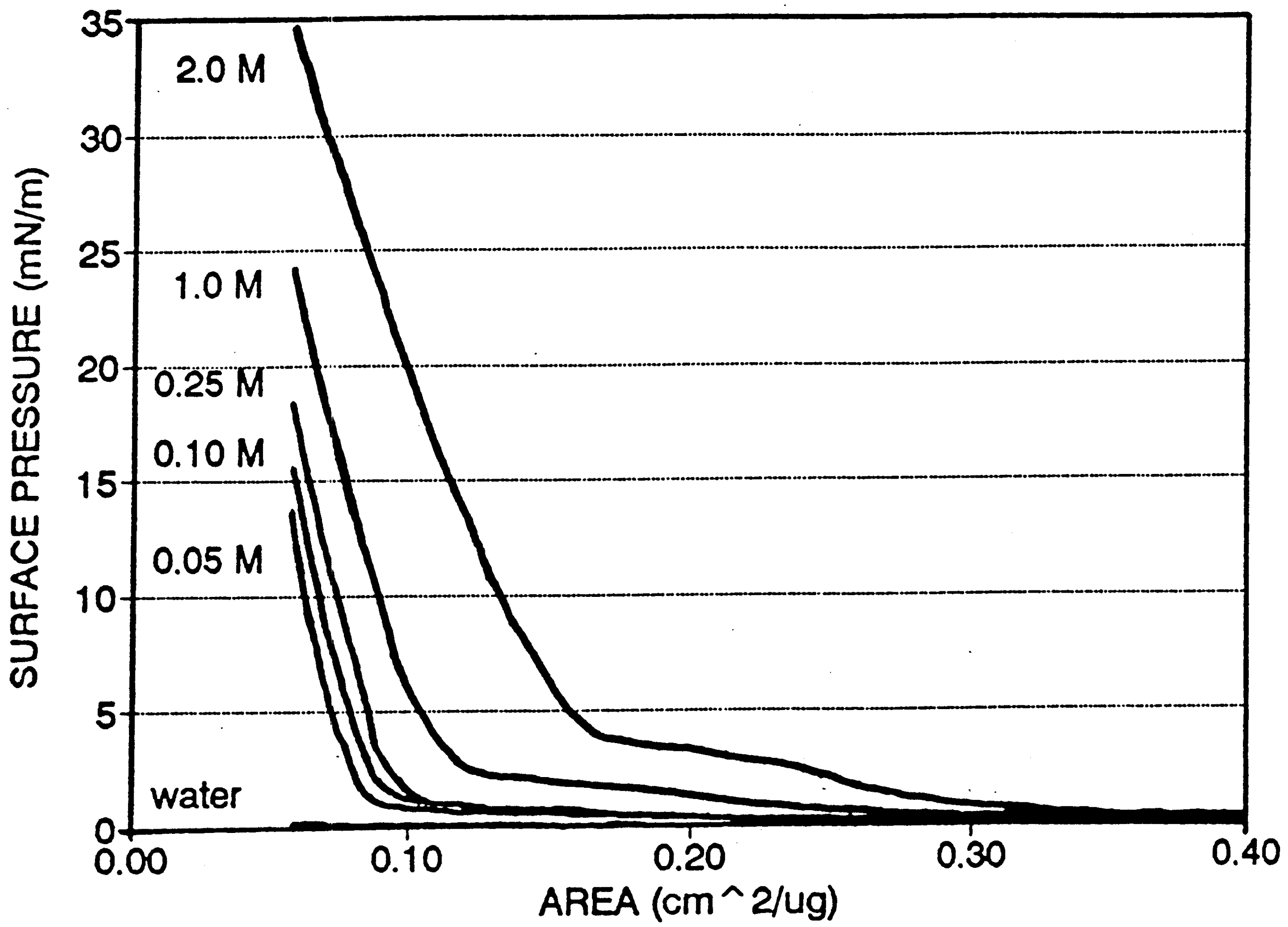
Table 3. Apparent molecular weight by the monolayer method, molecular weight by Size Exclusion Chromatography and surface fraction f.

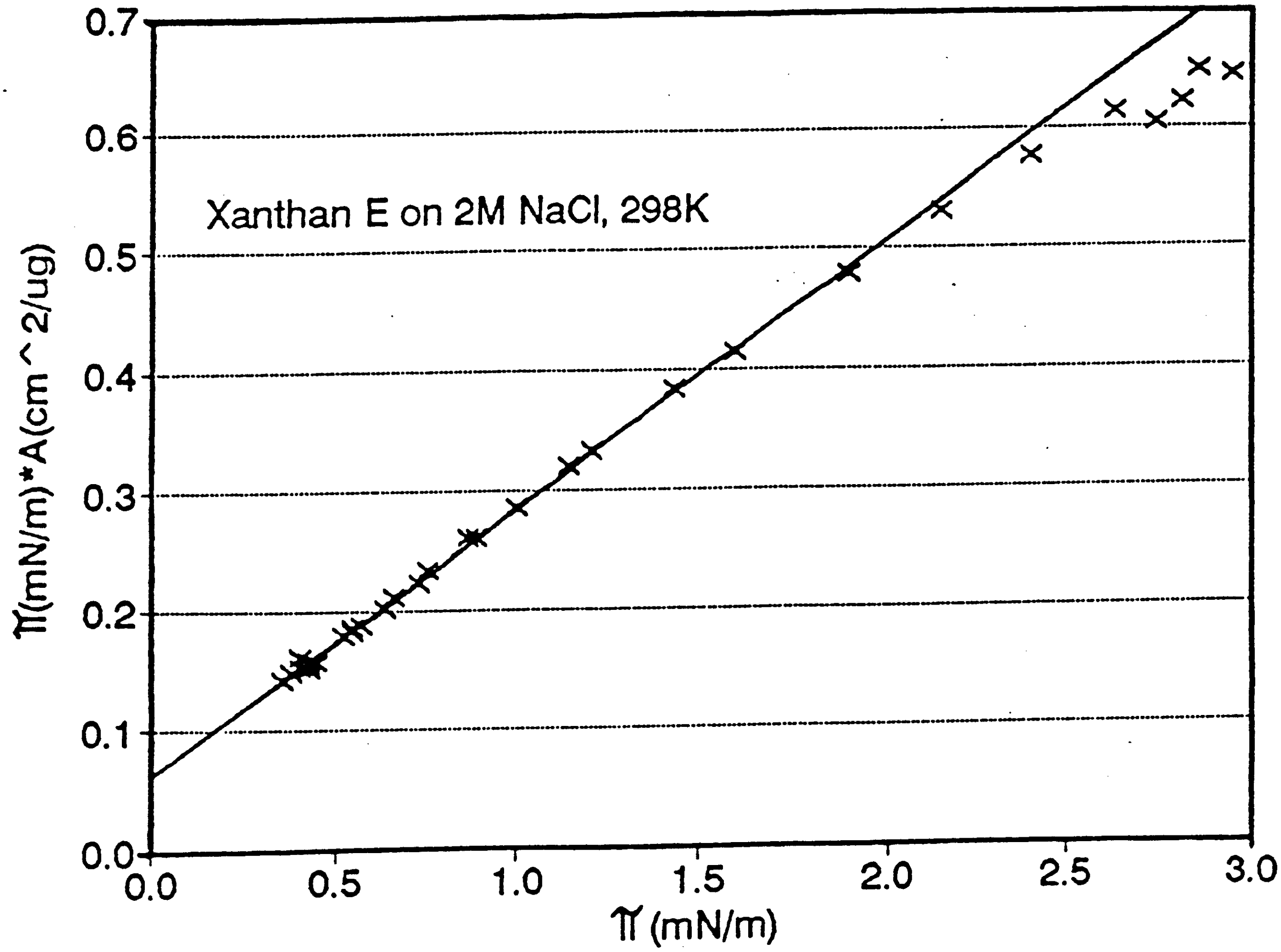
Sample	M(π-A)	M(SEC)	f	A
	10^5 g/mol			
Ap	20.8	127.1	0.16	
Bp	9.1	-	-	
Dp	2.9	-	-	
E	4.1	105	0.04	
F	9.3	-	-	
G	9.4	-	-	

Table 4. Effect of spreading media on initial spreading pressure π_s and final compression surface pressure π_f, 1000 ppm Xanthan E at 25°C on 2 M NaCl.

Sample preparation	π_s	π_f
	mN/m	
Dissolved in distilled water	0.21	30.0
Dissolved in distilled water added NaCl to 2 M NaCl	0.04	6.5
Dispersed in 2 M NaCl Aggregates in solution	0.02	16.2


XANTHAN E IN DISTILLED WATER
SURFACE PRESSURE (mN/m)
25
20
15
10
5
0
25
35
45
55
65
75
85
TEMPERATURE (C)


SURFACE PRESSURE (mN/m)
35
30
25
20
15
10
5
0
2.0 M
1.0 M
0.25 M
0.10 M
0.05 M
water
0.00
0.10
0.20
0.30
0.40
AREA (cm ^ 2/ug)


Xanthan E on 2M NaCl, 298K
Π(mN/m)*A(cm ^ 2/ug)
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
Π (mN/m)

Xanthan surface conformation