Rheological Properties of High Concentrations of Carboxymethyl Cellulose Solutions

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ABSTRACT: The study was concerned with measurements of steady-state parameters, the transient shear stress response, and the yield stress of carboxymethyl cellulose (CMC) solutions at high concentrations. Tests also included thixotropic and viscoelastic behavior and dynamic responses. The concentrations ranged by weight from 5 to 8% of CMC. The steady-state shear flow showed that at higher shear rates the viscosities of CMC solutions tend to be less dependent on the concentration. The solutions showed rheopectic behavior for very small shear rates. No yield stress was detected. Measurements recorded the thixotropic behavior. At higher stress values, nonlinear viscoelastic effects were detected. Dynamic viscosities measured in a dynamic test were higher than were the shear viscosities at the same concentrations. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 79: 1787–1801, 2001

Key words: carboxymethyl cellulose; rheological study; viscosity; viscoelasticity; yield stress

INTRODUCTION

Water-soluble carboxymethyl cellulose (CMC) is a white granular substance available at various levels of viscosity (10–50,000 mPa s in 2% solution) and is equally soluble in hot and cold water. It is the sodium salt of CMC, having a loose number of sodium carboxymethyl groups (CH_2COONa) introduced into the cellulose molecule to promote water solubility. On the other hand, CMC is the cellulose derivative, which is the most widely used. It is made by treating cellulose with sodium hydroxide–chloroacetic acid. The single most important property of CMC is viscosity building. Each polymer chain in a diluted solution of CMC is hydrated and extended and exhibits a stable viscosity. However, the presence of metal salts has little effect on the viscosity. CMC solutions are either pseudoplastic or thixotropic depending on the type, but most of them are highly pseudoplastic. They also show some thixotropic behavior, that is, the viscosity decreases gradually by shearing. CMC is manufactured by many companies throughout the world and has a large market in Western Europe and North America. The properties of CMC along with its versatility as a thickener, film former, protective colloid, and water-retaining agent have made CMC the most produced and widely used industrial cellulose ether.

Large quantities of CMC are produced in crude commercial grades without any refining for use in oil drilling, detergents, and the paper industry. In the oil industry, CMC is used to increase the stability of mud for drilling purposes as it stabilizes the aqueous suspensions of clay due to its high water retention. CMC can be useful as a soil stabilizer, in detergent composition, and as a tex-

tile surface protector. In the paper industry, CMC also acts as a strengthener and binder and increases the water retention of the coating mixtures, thereby increasing grease resistance and the print capability of paper. Pigment coats containing purified CMC are applied on the size press and can be up to 10% by weight. High-purity grades are employed as food additives. In the food industry, it is added as a thickener or as an emulsion stabilizer to some products such as frozen dairy, dry drink mixes, icing, syrups, and baked goods. CMC works as a bio-adhesive agent to prolong the expiration period of drugs.

CMC has been the topic of much recent research.[1–7] Sikkema and Janssen[4] attempted to prepare CMCs with properties similar to xanthan gum solutions. They manipulated the alkalinity during CMC preparation and produced a complex CMC colloid fiber. Westra[5] studied the rheological properties of an aqueous solution of a cellulose–CO–CMC segmented block copolymer that had been synthesized. The study revealed properties that are similar to those found in highly pseudoplastic solutions of xanthan gum. Young and Shoemaker[3] measured the intrinsic viscosities of aqueous suspensions of xanthan gum and CMC by using a cone-and-plate rotational rheometer. The authors concluded that the reduced viscosities of CMC suspensions and xanthan gum suspensions within the concentration range of 0.04–0.01% increased with the dilution. Abdelrahim et al.[2] stated that they had focused their study on measuring the effects of the concentration and temperature on the CMC rheology. They chose to study the low range of CMC concentration ranging by weight from 0.5 to 2% over a temperature range of 30–90°C. A Haake Model RV20 rotational viscometer was used to apply a programmed 3-cycle continuous sequence shear rate. They concluded that the power law model was best for describing the flow behavior of CMC and that the power law parameters $\{m, n\}$ were sensitive to the change in concentration and temperature. Dolz-Planas et al.[6] analyzed the thixotropy of CMC gels at different concentrations ranging by weight between 1 and 2.5%. The analysis was in terms of the concentration, duration of storage time, agitation time, and temperature. The thixotropic area variation rate was defined for determining the structural breakdown rate. They noted that the variation rate became negligible after 5 min of agitation and was independent of the rest of the remaining variables. In another publication by Dolz et al.,[7] a general method for quantifying the thixotropic behavior of systems with low thixotropy was proposed. This method was applied to CMC gels with high viscosities. Ghannam and Esmail[1] carried out a complete and comprehensive rheological study of low concentrations of CMC solutions using the Haake-Rheostress RS100 system. It was determined that the rheological behavior was transformed from Newtonian to shear thinning as the concentrations of the solutions increased.

At different concentrations, CMC has a wide range of diverse applications such as in the production of drilling mud and detergents, in the paper industry, in food products, and in pharmaceuticals. The objective of this article was to provide a complete and comprehensive rheological study of a high range of CMC concentrations. Throughout the testing process, a Haake-RS100 cone-and-plate sensor system was used. We analyzed the rheological behavior of CMC gels at concentrations of 5, 6, 7, and 8% by weight. The thixotropic area and its variations with factors such as concentration and agitation time were obtained. The rheological fingerprints of the samples provided the assigned shear rate and the resulting shear stress correlation in the controlled rate mode of the rheometer.

EXPERIMENTAL

CMC–sodium salt (medium viscosity), No. C4888, with a degree of substitution (DS) = 0.7, was purchased from the Sigma Chemical Co. (St. Louis, MO). It is a completely water-soluble polymer and not an ion exchanger, with a viscosity of 2% aqueous solution at 23°C: 400–800 mPa s. The solutions were studied at concentrations of 5, 6, 7, and 8% (w/w). Each hydrogel concentration was prepared by adding a specific weight of CMC to 0.5 L of distilled water. Stirring time was required for the gels to be homogenized and this was done at a room temperature of 23°C. The experimental measurements were conducted after the samples had been maintained at the same temperature for a sufficient time to achieve complete dissolution. The concentrations of CMC chosen for this study allow for full coverage of the high concentration range, within the limits of the test equipment.

The RS100 rheometer is usually used to determine the characterization of viscoelastic fluids in both research and quality control. It features an alternative controlled rate (CR) and oscillation
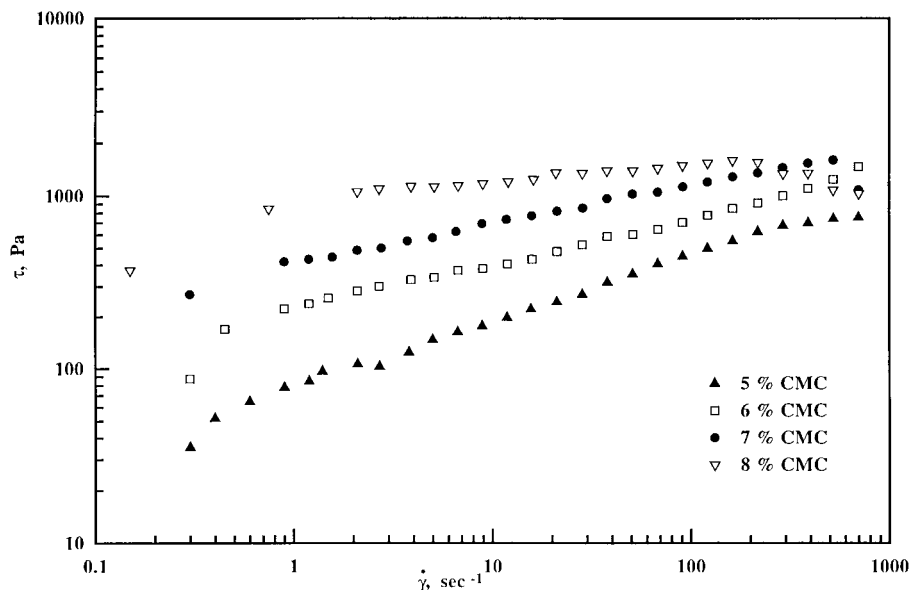
Figure 1 Flow behavior curves of CMC solutions.

(OSC) test modes, and it is designed with a special low friction roller bearing for the rotor shaft that allows for the testing of fluids at low and high shear rates. Extremely low inertia (shear stress) can be applied on the samples tested by the rheometer-controlled stress mode. Easy optimization of test parameters can suit many samples with different rheological behavior by the interchangeable sensor systems. The RS100, with its digital encoder capable of processing 1 million per revolution, can detect any deformation of the samples. Very small yield values and very low strains or shear rates can be measured due to the high rheometer encoder resolution. The rheometer has a computer-controlled system that provides oscillating stress inputs and autostrains as well as swapping between both the CS and CR modes. It also controls the variable life speed, which axially positions a cone versus plate, manually or remotely. A thermal gap size compensation for any of the sensor systems is provided by a microprocessor–control. Moreover, the thixotropy of samples by using the hysteresis between the up and down ramp curves can be determined by the CR mode.

RESULTS AND DISCUSSION

Steady-state Shear Flow

In this test, two main relationships of double logarithmic scales illustrate the rheological finger-prints of the samples under study. These are the flow behavior curves (Fig. 1), showing the relationship between shear stress τ and shear rate γ and the viscosity curves (Fig. 2) which illustrate the viscosity η variation with a gradual increase in shear rate. Figures 1 and 2 are based on the assigned shear in CR mode ranging from 0.15 to 700 s^{-1} and the resulting shear stress for CMC solutions in the concentration range of 5–8%. All samples seem to show a strong pseudoplastic behavior. A concentration of 5% CMC compares well with the same measurements reported by Ghannam and Esmail.[1] Figure 2 shows the shear thinning which is becoming much stronger for the higher concentration. At higher shear rates, the viscosity breakup is quite evident for all tested concentrations. Viscosity is in the range of 1,000,000–100,000 mPa s at low shear rates for the CMC concentrations of 5–8%. Viscosities of all concentrations approach the value 1000 mPa s as the shear rate tends to 1000 s^{-1}. *Moreover, at high shear rates, the viscosity of 7% experiences a strong drop. A similar drop is experienced by solutions of 8% concentration at a lower shear rate of 300 s^{-1}.* Linear correlations of log (η) and log (τ) with log (γ) exist. These relationships were modeled by the Ostwald-de-Waele equation (power law), which is known to be a suitable model in this case. The equation can be written as
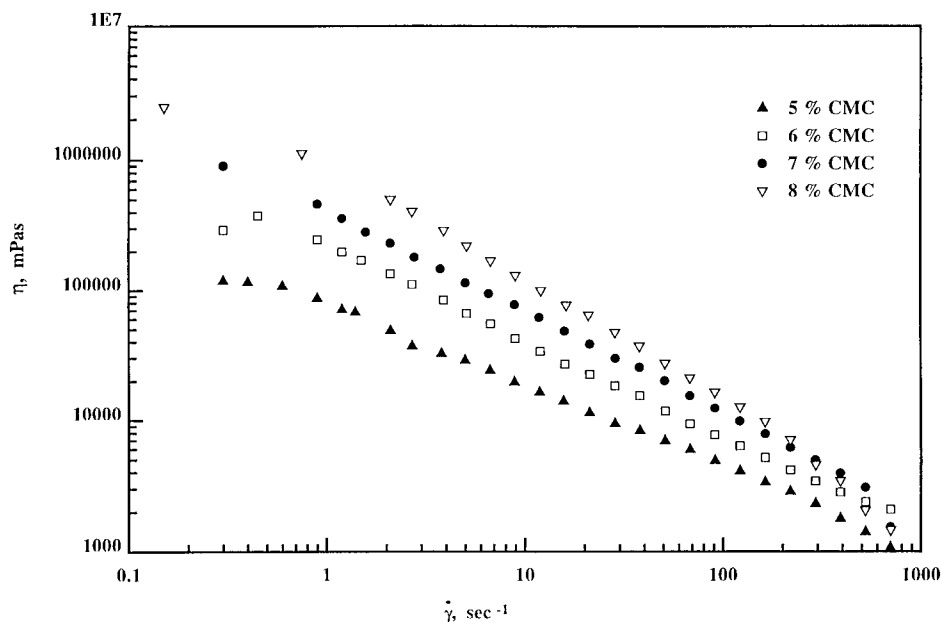
$$\tau = k \ (\gamma)^n \tag{1}$$

Figure 2 Viscosity curves of CMC solutions.

where τ is the shear stress (Pa); γ, the shear rate (s^{-1}); k, the consistency index (Pa s''); and n, the flow behavior index. Table I shows both the k and n constants of the Ostwald-de-Waele equation for concentrations from 5 to 8% CMC and indicates the drop of flow behavior index from $0.38-0.092$, which is a clear indication of the strong pseudoplastic behavior of CMC concentrations above 5%.

Transient Shear Stress Response

Transient rheological behavior is a very characteristic time effect, leading to a complex relationship between the stress and the rate of deformation. For polymeric liquid and concentrated solutions, the overshoot can be explained in terms of a transient entanglement network. These entanglements are the local interactions between the macromolecules that are continuously being destroyed and reformed at rates depending on the

Table I Ostwald-de-Waele Equation Constants of CMC Solutions

CMC (%)	k	n
5	75	0.38
6	200	0.28
7	410	0.21
8	880	0.092

deformation field. In linear viscoelasticity, that is, when the deformation is slow or small, the stress function grows until a steady state is reached and it is strain-rate-independent. If the rate of deformation is large enough, the equilibrium entanglement density becomes strain-rate-dependent, and nonlinear viscoelastic behavior is observed. Initially, the stress growth function follows the curve of linear viscoelasticity until a critical strain (γ_{max}) is reached where maximum stress is observed.

In a Newtonian liquid, applying a constant shear rate leads to the beginning of immediate flow. From the rheological results of the graphs in Figures 3–6, one can easily notice the slow developing shear stress at low shear rates in different time intervals and that this interval becomes much shorter if the shear rate is increased. After this time-interval constant, the shear stress response is reached and the flow is called steady when the shear stress continues to remain constant with no inertial effect and drops to zero when flow is stopped. Transient behavior time can be long and comparable with the flow duration.

In this work, the transient response test was carried out with the RS100 rheometer. The controlled rate (CR) mode and time-curve option for a duration of 5 min for each shear rate was applied on the samples. The shear rates ranged from 0.15 to 500 s^{-1} for each CMC concentration. These
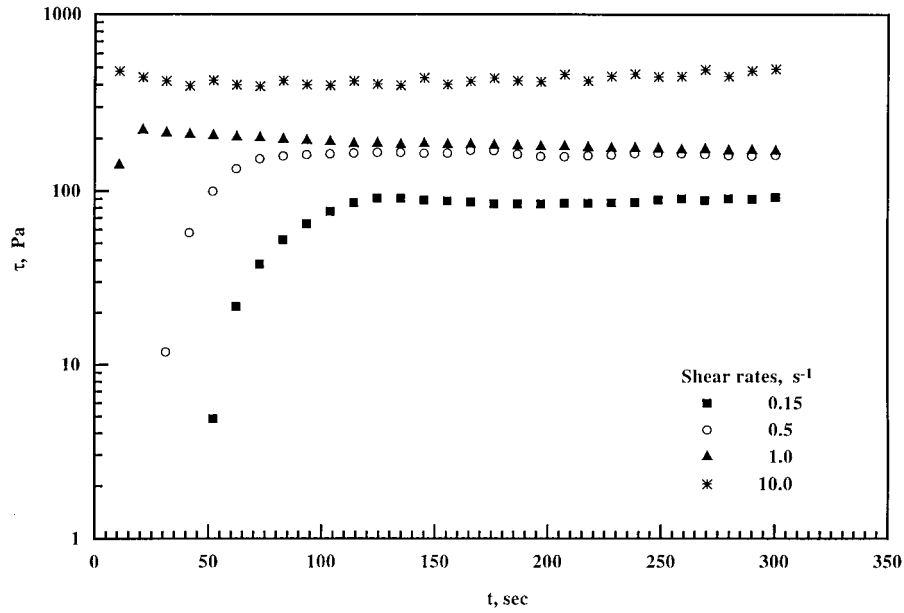
Figure 3 Transient shear stress response of 5% CMC.

tests show the existence of the rheopexy behavior, which is the time dependency of shear stress. Shear stress increases rapidly to a constant value for each concentration tested. Low and high CMC concentrations at shear rates higher than 10 s^{-1} do not exhibit rheopexy as proved by the test. The gradual increase or decrease in the resulting shear stress can be attributed to the structure

recovery. The qualitative rheopectic behavior is similar for all concentrations. The steady-state value of stress converges to 1000 Pa for all values of the shear rate, with increase in the concentration. The control concentration of a 5% CMC solution shows a response which compares well with the one reported by Ghannam and Esmail[1] for low shear rates.
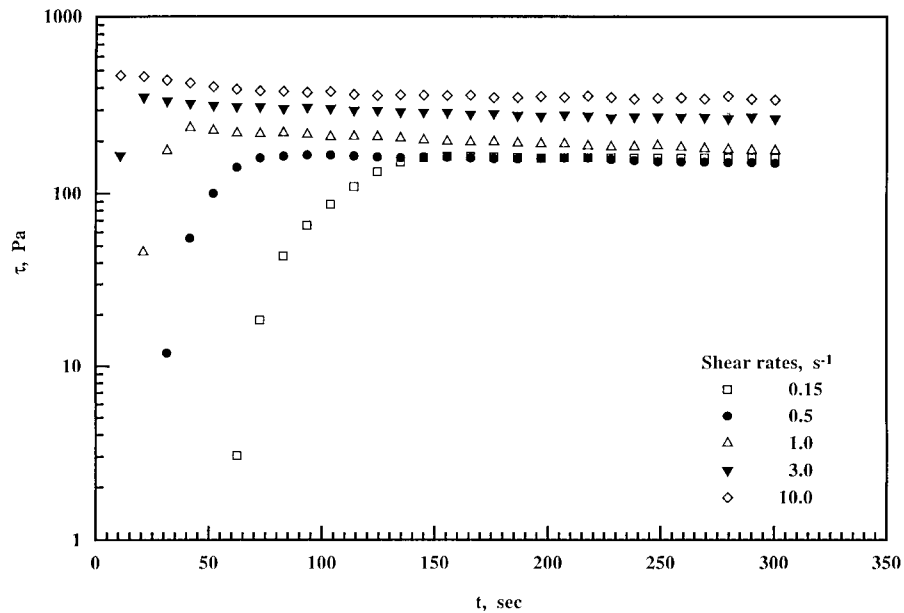

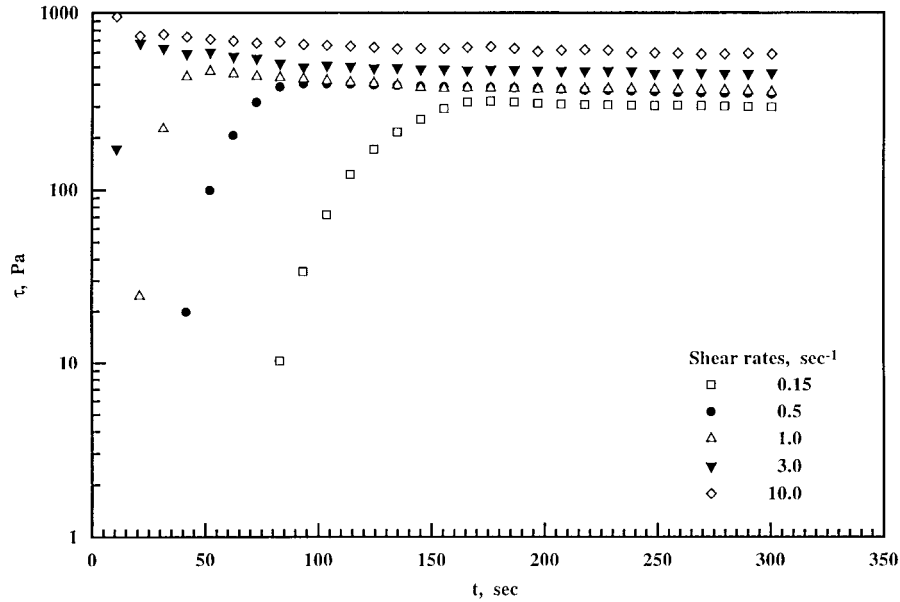**Figure 4** Transient shear stress response of 6% CMC.

Figure 5 Transient shear stress response of 7% CMC.

Yield Stress

Yield stress is the stress corresponding to the transition from elastic-to-plastic deformation τ_y (Pa) or the limiting stress below which a sample behaves as a solid. Also, it can be defined as the elastic deformation that disappears when the applied stress is released. In general, the yield point is the stress applied that causes unlimited deformation where the sample starts to flow. In this study, the yield test was performed using the RS100 rheometer in a controlled shear stress mode and the stress ramp option with 180 s per segment. The test was conducted by applying two segments of gradual increase, followed by an immediate decrease in shear stresses in three different tests. These segments were 0.09 Pa–4 Pa–0.09 Pa, 0.09 Pa–50 Pa–0.09 Pa, and 0.09 Pa–100 Pa–0.09 Pa for 180 s for each segment. These
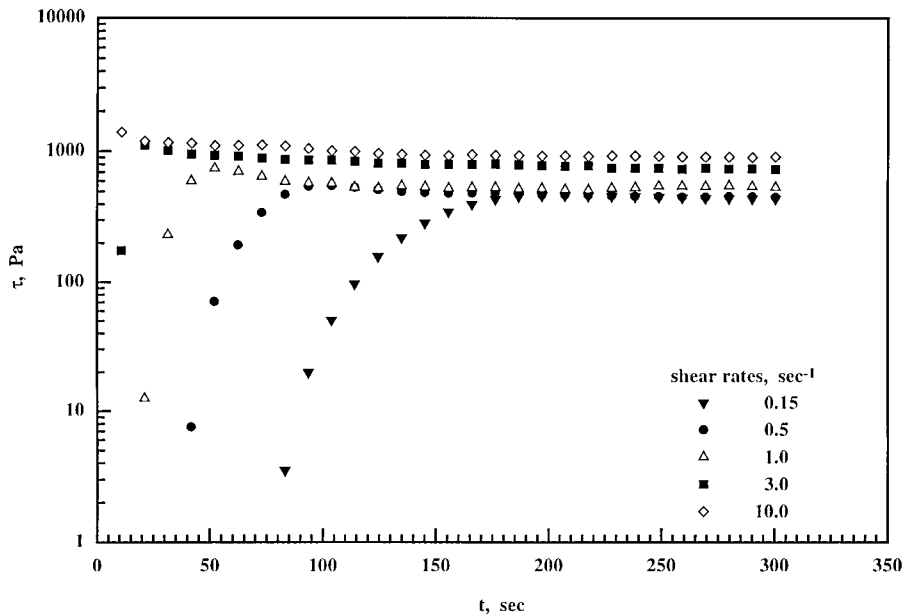

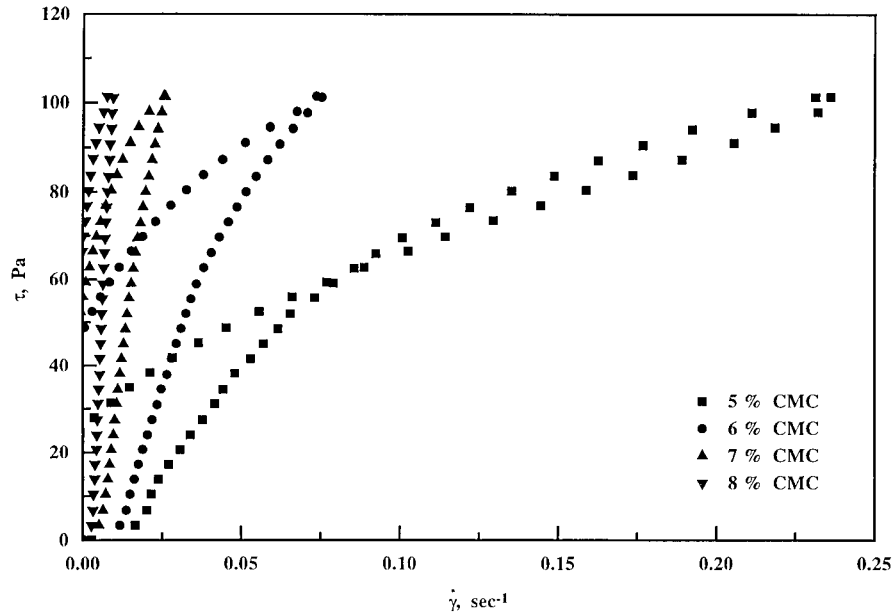**Figure 6** Transient shear stress response of 8% CMC.

Figure 7 Yield stress response of CMC at (0.09 Pa–100 Pa–0.09 Pa) segments.

three separate tests were performed to detect any possible deformation behavior for the concentrations of CMC of 5–8%. Yield stress was analyzed on the resulting plots of τ versus γ, which are defined by conclusions from the literature.[7–9] Figure 7 shows the yield stress response for all the concentrations used. The test shows that higher concentrations of CMC produce a higher resistance to flow even though the solutions showed no yield stress.

Thixotropy Test

Thixotropy of a material can be defined as the tendency of a substance to change from a gel state with high viscosity to a lower viscosity as a result of applying a high shear rate. An important characteristic of thixotropy is its capacity to rethicken when the material is left at rest. The practical use of materials in paints, coatings, adhesives, etc., rely upon the properties of viscosity such as the increase in the rested state and its decrease in the case of applying a constant shear rate. Moreover, such materials are time-dependent fluids, which means that their thixotropic structure can be broken down in a matter of seconds, but it takes hours to fully recover to a gel state. In paints, for example, full recovery to a gel structure is needed as fast as possible to prevent the paint layer from sagging off the wall.

The thixotropy test was carried out using the RS100 rheometer in the mode of a controlled shear rate, where the shear rate is programmed to increase from 0.15 s^{-1} to its maximum value of 700 s^{-1} to give the up curve. The down curve is immediately ramped down from 700 s^{-1} shear rate to 0.15 s^{-1} in the same time as the up one. CMC concentrations of 5–8% were used in the test samples to study any possible thixotropic behavior as a function of agitation time and concentration.

Two different techniques have been introduced in this test to check the behavior. The first technique involved agitating the sample by 60, 100, and 200 s for each curve of the up-and-down cycle. This procedure was applied for CMC sample concentrations of 5–8%. Also, agitation times of 60, 100, and 200 s that were programmed in the CR mode as a test of three constant time parts. A shear ramp of 0.15–700 s^{-1} was maintained in one test; then, the shear rate was kept constant in the second part as time curve at 700 s^{-1}. Finally, in the third part, it was allowed to ramp back from 700 to 0.15 s^{-1}. This procedure was followed in the second technique of the thixotropy test for all tested CMC concentrations.

All the CMC concentrations used in the study exhibited a thixotropic effect in both techniques. This confirms the transient test responses, which demonstrated earlier that the samples are time-dependent material. Furthermore, the conclusion of a steady-state test, that the high CMC solutions revealed a shear thinning or pseudoplastic

Table II   Thixotropy Hysteresis Areas (kPa/s) for the First Technique: "One Cycle of Up and Down Curves"

Time	Concentration			
	5%	6%	7%	8%
60 s	96	135	328	526
100 s	81	122	269	410
200 s	78	119	222	317

Table IV   Thixotropy Hysteresis Areas (kPa/s) for the Second Technique: "The Cycle of Up–Constant–and Down Curves"

Time	Concentration			
	5%	6%	7%	8%
60 s	151	245	493	550
100 s	146	212	380	470
200 s	144	195	231	387

material behavior, is confirmed by showing the thixotropic response in this test. Results from the first and second techniques of the thixotropy test reveal the existence of variation in the thixotropic areas for the CMC concentration with shearing time. Obviously, by comparing the results obtained in Tables II–IV and in Figures 8 and 9, one can determine that, for a given shearing time, the thixotropic area of the CMC gels increases proportionately with the CMC concentrations used. In evaluation of Figures 8 and 9, which show the relationship of the obtained thixotropic hysteresis areas in kPa/s with the time of agitation in seconds and CMC concentration, the areas are higher for the higher concentrations which are characterized by a significant increase in their viscosity. Moreover, for each curve in Figures 8 and 9, the area decreases with increase of the shearing time for all CMC concentrations and this decrease becomes sharper with increase in the concentration.

The conclusion of the thixotropy test is that there is an obvious buildup of areas of hysteresis with increased concentration. Large thixotropic areas were obtained due to a large increase in the CMC viscosity with increase of the concentration. Earlier, we concluded that transient test results confirmed those of the thixotropy test that the CMC material is time-dependent and pseudoplastic. The area decreases with increase of the shear-

Table III   Thixotropy Hysteresis Areas (kPa/s) for the First Technique: "Three Cycles of Up and Down Curves"

Time	Concentration			
	5%	6%	7%	8%
200 s	142	209	340	674

ing time for all CMC concentrations and this decrease becomes sharper with increased concentration. The yield stress test of the solutions confirmed the existence of the thixotropy behavior although no yield stress was detected.

Creep Recovery Test

Another important aspect of the rheological characterization of CMC solutions in higher concentrations is the creep-recovery property. Creep is the test where a constant stress is applied on a sample and the resulting strain is obtained. It is also defined as a slow development of deformations, $\gamma(t)$, at that applied constant stress. Time-dependent compliance, $J(t)$, is introduced as the material constant in the creep equation:

$$\gamma(t) = \tau\, J(t) \tag{2}$$

For the given applied stress, the higher the material compliance, the easier it can be deformed. The recovery part of the test is a measure of the decline of the material deformation when the stress is removed. Recovery effects can strongly enhance the material quality. A slow recovery is an indication of the ability of the material to store the residual stress.

Creep-and-recovery experiments were carried out using the Rheometer RS100 at a controlled stress mode at various stress levels ranging from 0.02 to 150 Pa. The test consisted of two main parts—creep and recovery. These parts were performed on the same sample for a period of 300 s each. The objective of the test was to distinguish between the linear and nonlinear viscoelasticity of the material under study. In the linear viscoelastic region, the compliance of the material is independent of the applied stress. Moreover, the material network structure is elastically deformed and it can recover only when the stress is
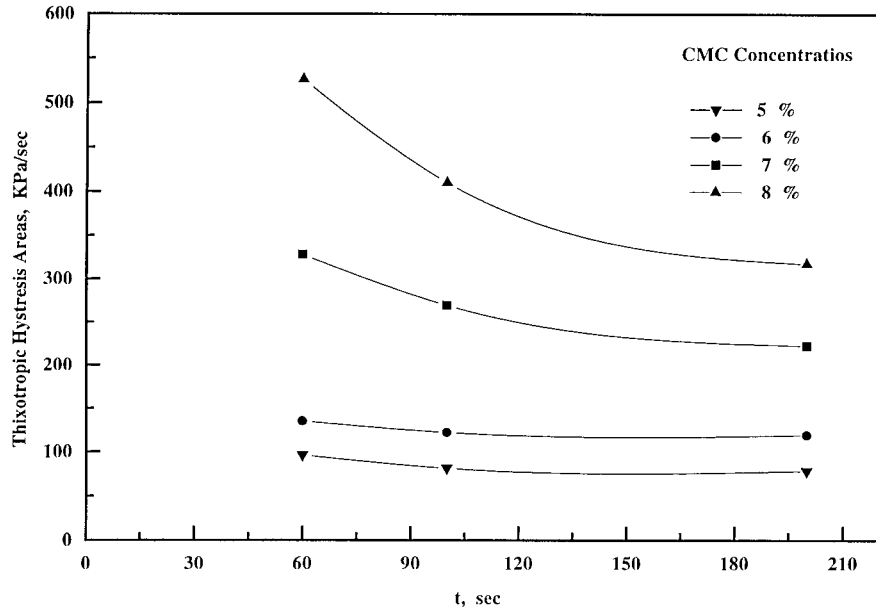
Figure 8   CMC thixotropic hysteresis areas by the first technique.

removed. On the other hand, higher stress values lead to the nonlinear viscoelasticity and the material is continuously deforming, that is, it flows. In a previous study by Ghannam and Esmail,[1] it was shown that CMC at low concentrations has a viscoelastic characterization. They found that for a concentration of 3% the linear viscoelastic range has a limit of 10 Pa of stress and the response was nonlinear for 15 Pa.

The creep-recovery results of the solutions of 5–8% CMC are plotted in Figures 10–12. The results indicate that CMC solutions at high concentrations continue to behave as viscoelastic materials. Elasticity is seen to be lower for all the solutions with increased stress. This is due to the structural breakdown that occurs. The recovery part of the samples showed that the strain has less recovery for increased values of shear stress.
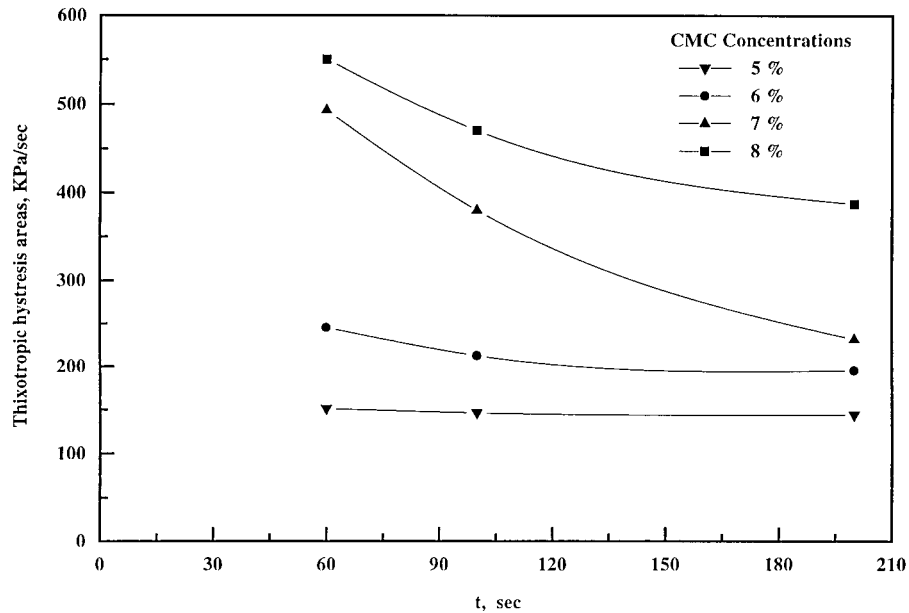

Figure 9   CMC thixotropic hysteresis areas by the second technique.
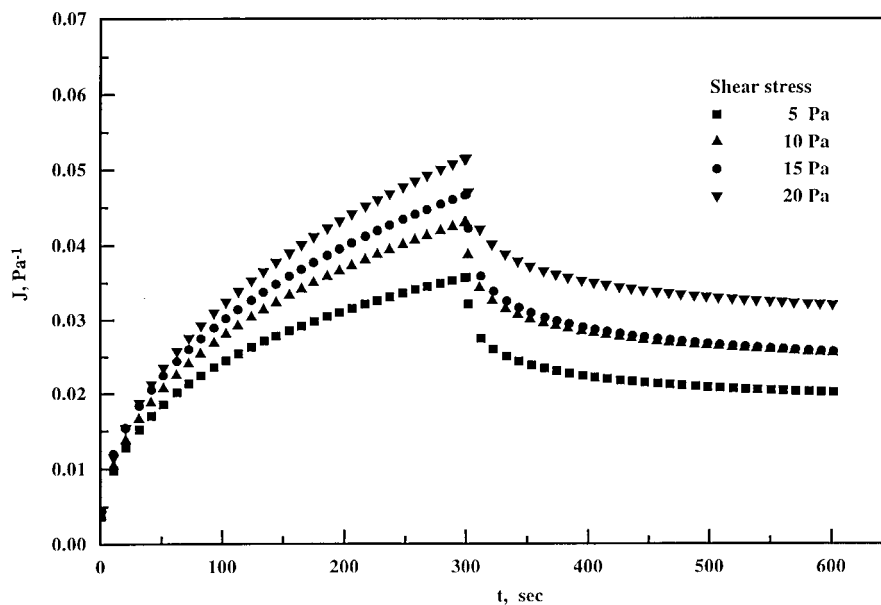
Figure 10 Creep-recovery curves of 6% CMC.

This can be clearly seen in the plots of 8% CMC concentration at stress values of 1, 50, and 150 Pa. Viscoelastic properties of the solutions can be better analyzed at lower values of stress where the deformation occurs with insignificant or little breakdown in the material structure. Generally, all the results of this test can be categorized in two ways: An instantaneous shear stress is constantly maintained for 300 s, and the response is

the instantaneous increase of the strain or deformation. When the stress is removed, an instantaneous component of the strain recovery is evident in the plot. An elastic response or time-dependent recovery follows this in 300 s. It has been found that a period of 10 min divided between the creep and the recovery parts is sufficient for analyzing the linear viscoelastic behavior of the given samples. A high stress applied to the sample led to a
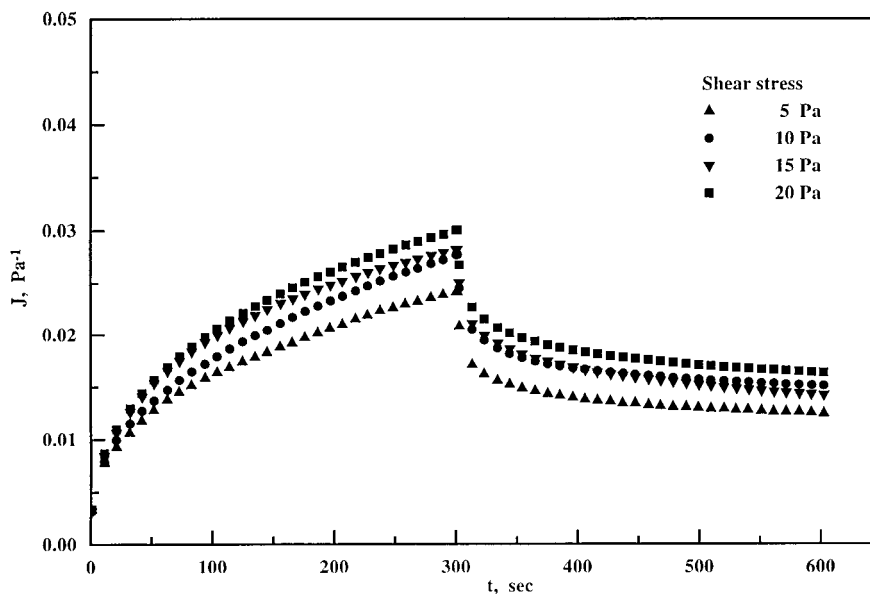

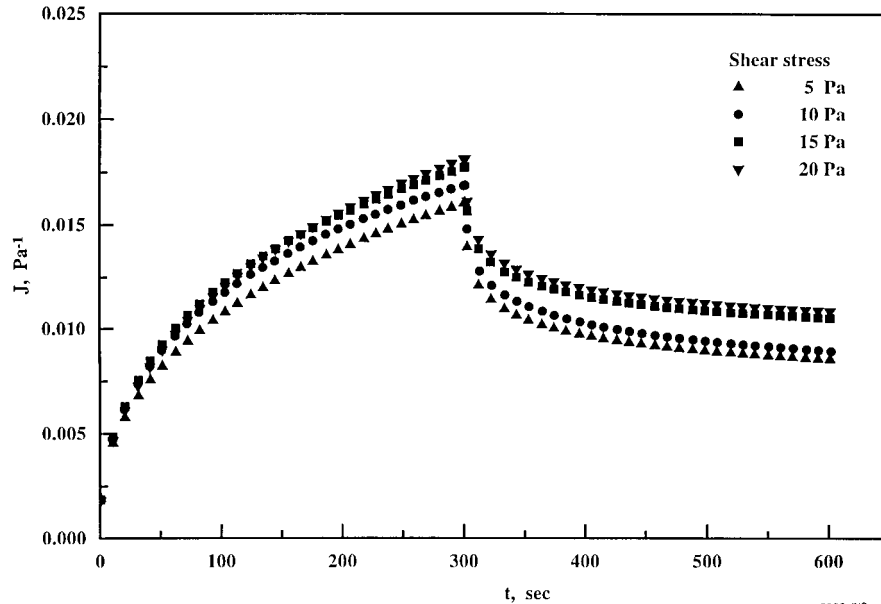**Figure 11** Creep-recovery curves of 7% CMC.

Figure 12 Creep-recovery curves of 8% CMC.

breakdown in the structure and a material flow. A decrease in the curvature of the creep curve indicates a strong decline in viscosity, and recovery is slowed or absent at these stresses. An absence of recovery shows that the sample is in a viscous flow and that no elastic effects remain active.

The results in Figures 10–12 show a comparison between the linear viscoelastic ranges for the concentrations 6, 7, and 8% CMC at several constant shear stresses. The shear stresses chosen were 5, 10, 15, and 20 Pa as plotted in the three graphs. The linear viscoelastic range for the creep curves at constant stresses converges with the increase in the solution concentration. On the other hand, when a higher stress such as 50 Pa is applied for the solutions of 5 and 6% CMC, the compliance curves indicate a nonlinear viscoelastic effect.

Dynamic Test

Dynamic tests are an extremely useful tool to study and evaluate the viscoelastic behavior of polymeric materials. In this study, they were used to detect some aspects of the viscoelasticity of CMC solutions that cannot be described in the creep-recovery tests. The tests were carried out using the Rheometer RS100 in the oscillating mode. Two different tests were performed on the CMC solutions in this work: the stress sweep test and the frequency sweep test. Linear viscoelasticity prevails for small amplitude oscillatory shear,

in which the polymeric structure is not significantly disturbed from its equilibrium state, and the material functions are independent of the amplitude of deformation. The strain and the resultant stress can be expressed as follows:

$$\gamma = \gamma_0 \sin \omega t \tag{3}$$

$$\tau = \tau_0 \sin(\omega t + \delta) \tag{4}$$

where γ_0 is the strain amplitude; τ_0, the stress amplitude; δ, the phase angle; and ω, the angular velocity $= 2 \pi f$.

The amplitude ratio, τ/γ ($G^\bullet$), which is the total resistance of a substance against the applied strain, will be dependent on the strain amplitude in the case of high levels of applying the strain amplitude. Also, the linear viscoelastic material functions cannot be determined.

Stress sweep is the dynamic test used to determine the linear viscoelastic range. In the test, the limit between the linear and the nonlinear viscoelasticity is plotted as the complex modulus, $G^\bullet$, versus the applied stress. A fixed frequency of 0.1 Hz is used in the stress ramp mode in three different tests. In the first test, a stress ramp of 0.1–5 Pa is applied on all the solutions. Another two stress ramps of 0.1–30 Pa and 0.1–100 Pa are performed to cover the possible viscoelastic linearity responses in three regions of stresses: low, medium, and high. The linear viscoelasticity is
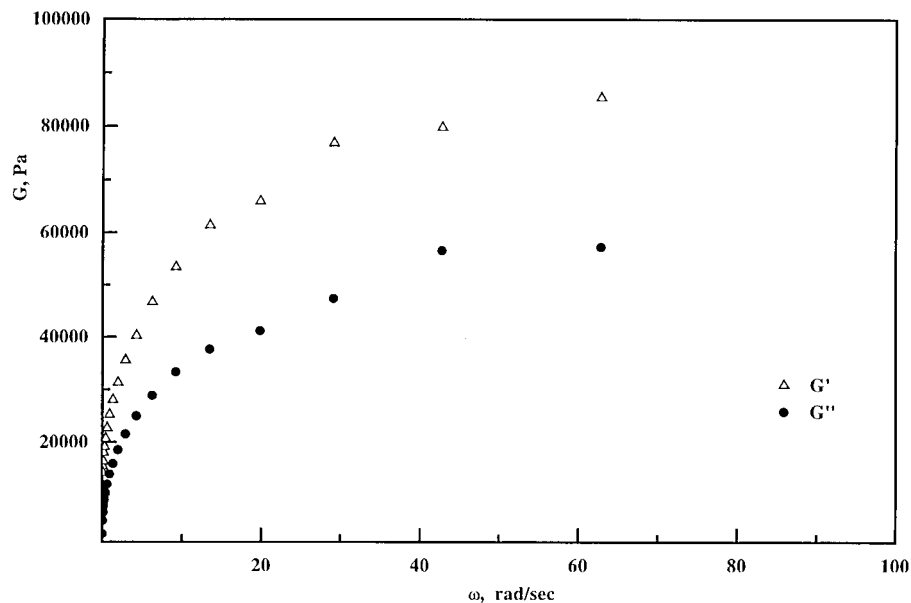
Figure 13   Elastic, G', and viscous, G'', modulus for 6% CMC.

detected when the $G^{\bullet}$ appears constant in a certain stress range. The nonlinearity is found to exist at high stresses where the sample could be deformed enough to destroy the internal temporary bonds of molecules. The results confirmed that the linear viscoelastic range for the 5–8% CMC samples is in the neighborhood of 15 Pa.

In the frequency sweep, the frequency ramp mode of 0.01–10 rad/s was applied to reveal the storage modulus, G', and loss modulus, G'', responses of the CMC samples within the linear viscoelastic range as plotted in Figures 13 and 14. The storage or elastic modulus and the loss or viscous modulus can be defined according to the equation
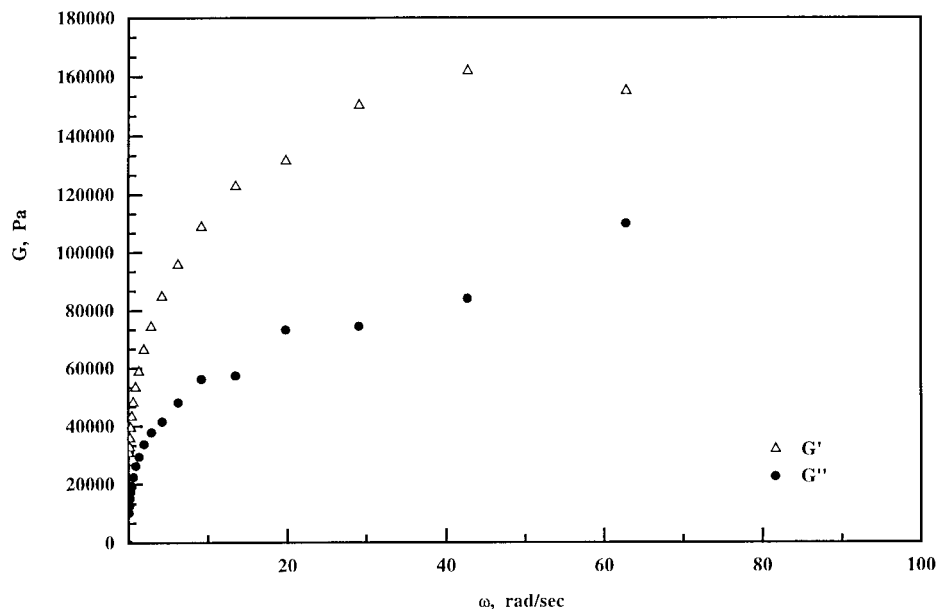
$$G^{\bullet} = G' + i\,G'' \qquad (5)$$

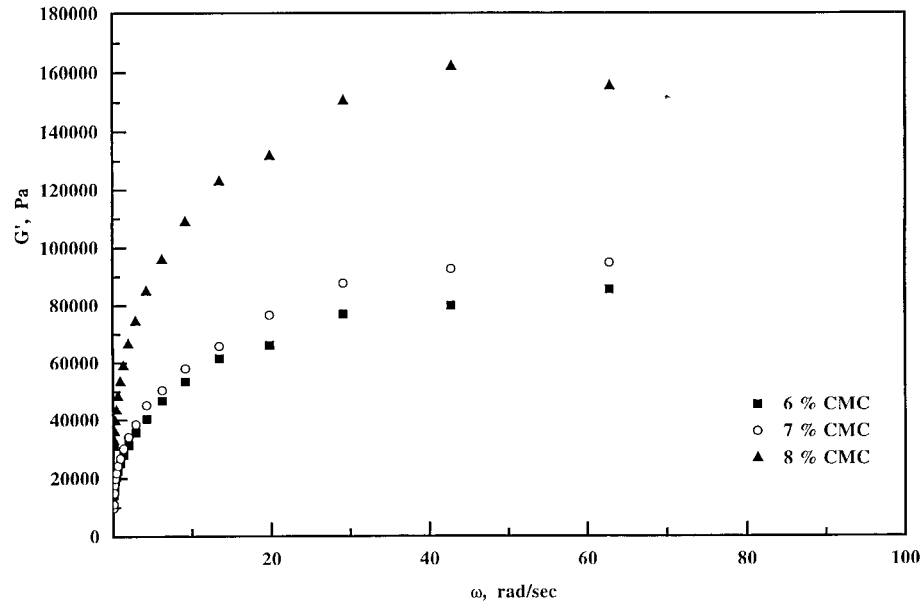**Figure 14**   Elastic, G', and viscous, G'', modulus for 8% CMC.

Figure 15 Elastic modulus of CMC solution.

where $G^{\bullet}$ is the complex modulus that is used in the stress sweep to determine the range of viscoelasticity. The results of the frequency sweep in Figures 13 and 14 show that the elastic behavior is higher than is the viscous one over the entire range of applied frequencies. In addition, the elastic modulus increases with increase of the concentration of CMC solutions as can be interpreted from Figure 15. The difference in the viscous mod-

ulus for CMC solutions described in Figure 16 is insignificant compared to the case of the elastic modulus.

The complex viscosity, $\eta^{\bullet}$, Pa s, is also obtained from the frequency sweep test. Figure 17 illustrates the complex viscosity behavior of the CMC samples versus the frequency that has been assigned in the test. Both dynamic viscosity and steady shear viscosity are known to be good char-
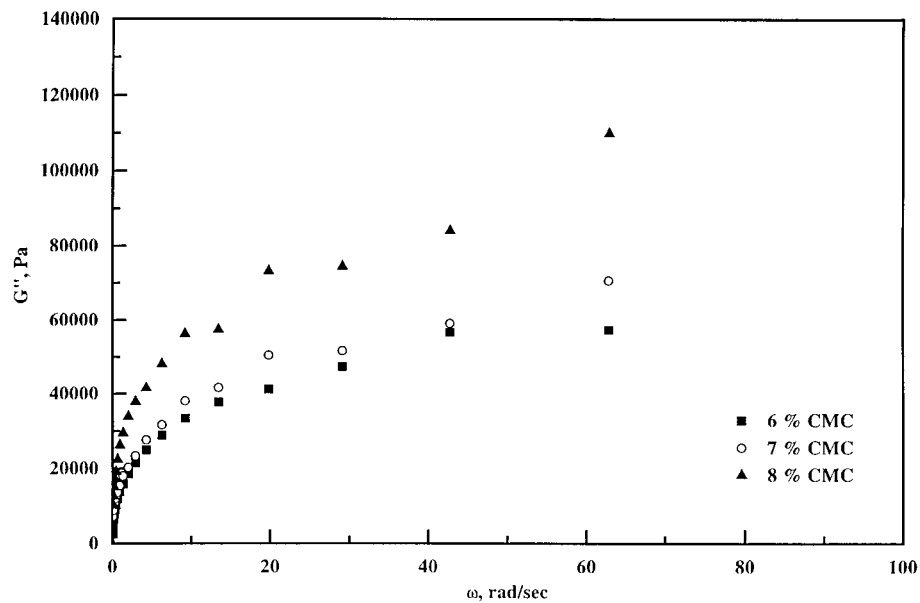

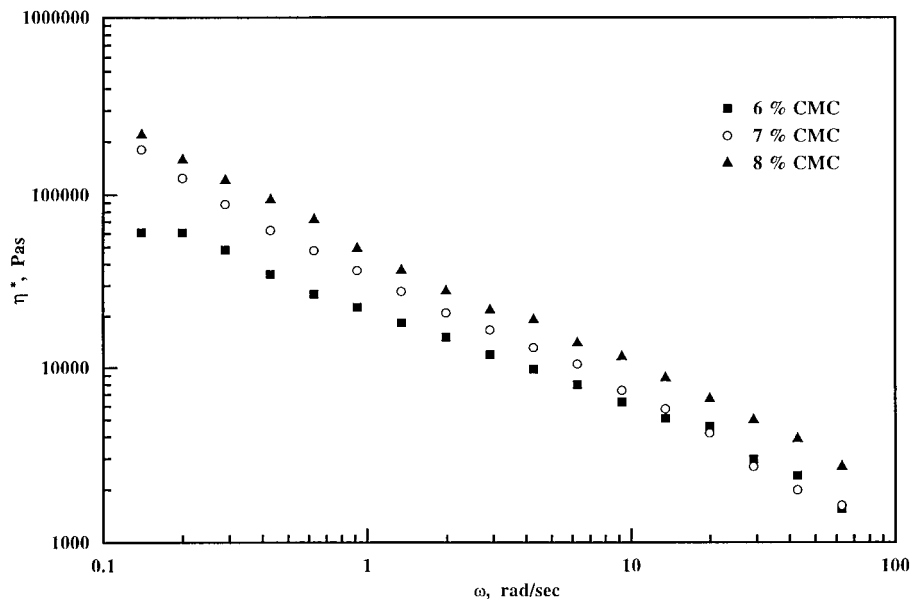**Figure 16** Viscous modulus of CMC solutions.

Figure 17   Complex viscosity for 6, 7, and 8% CMC solutions.

acterization tools in studying the rheology of some polymeric solutions. It is also known that they could have identical behavior. The shear and dynamic viscosities were not similar in the results of this work. This can be due to some intermolecular structure of this high range of concentrations of CMC. Dynamic viscosity decreases with the frequency for all CMC solutions, confirming the conclusion of the shear thinning effect detected in the steady shear viscosity test. Moreover, the complex viscosity, $\eta^{\bullet}$, is influenced significantly by the increase in the concentration of CMC. Shear viscosity for all concentrations is less than their dynamic viscosity as can be interpreted from both Figures 2 and 17.

CONCLUSIONS

CMC solutions starting from a 5% concentration showed a pseudoplastic or shear-thinning behavior for the entire shear rate range applied. Viscosity curves for 7 and 8% concentrations start to drop sharply at high shear rates of 500 and 300 s, respectively. It was found that the flow and viscosity curves mathematically follow the power law model and are represented by the rheological Ostwald-de-Waele model.

The transient shear stress response determined that the solutions studied showed rheopexy behavior or time dependency of the shear stress. Shear stress-overshooting behavior within the first 30 s of the test was found for the solutions when a shear rate of 100 s was applied. The thixotropy test results confirmed the ones that were detected in the transient test. The thixotropy behavior test showed that the area decreases with increase of the shearing time for all CMC concentrations and this decrease becomes sharper with the concentration increase. Also, increase of the CMC concentrations led to a buildup of hysteresis areas due to increase in the CMC viscosity. The yield stress test of the solutions confirmed the existence of the thixotropy behavior although no yield stress was detected, which also was in agreement with the conclusion of the same test response in another publication[1] for the lower range of CMC concentrations.

The creep-recovery results of the solutions of 5–8% CMC indicated that CMC solutions at high concentrations continue to behave as a viscoelastic material. The linear viscoelastic regions for the samples were in the neighborhood of 15 Pa. Around this value of stress, the creep curves converge with increase of the solution concentration. Dynamic tests produced results that came out with the same conclusion of the creep-recovery test. The results of the frequency sweep indicated that the elastic behavior is higher than is the viscous behavior over the entire range of frequencies for all the solutions. In addition, dynamic viscosity was found to be dependent on increase of

the concentration and to decrease by increasing the frequency for all CMC solutions. This confirmed the shear-thinning effect detected in the steady-state test.

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