Rheological Characteristics of a Detergent Slurry Containing Pentasodium Triphosphate

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The purposes of the present work are to analyze the rheological characteristics of a detergent slurry containing pentasodium triphosphate (TPP) and to study the influence that some factors exert on the rheological consistency of this slurry. Experimental results were achieved using a Brabender Plastograph and have been analyzed according to approximate methods to convert torque-rheometer data into fundamental rheological units.

From the experimental results it can be inferred that: (i) The systems studied exhibit plastic rheological behavior at steady shear and a progressive decrease in consistency by increasing shear rate; (ii) an increase in the yield value and apparent viscosity of the slurry are produced by increasing the concentration of nonaqueous substances or the phase I/phase II TPP ratio; and by decreasing the temperature or the hydrotrope content; (iii) the effect that temperature and concentration exert on the rheological consistency of the slurry may be quantified according to the usual equations found in the references.

Properties of detergents depend upon the concentration of Ca^{++} and Mg^{++} ions in the washing medium (1, 2). The action of these ions can be avoided by sequestration or precipitation. Hence, a product acting as a builder must be included in the detergent. Classically, the most widely used of these is pentasodium triphosphate (TPP), which appears in two crystalline anhydrous forms called phase I and phase II.

The influence that TPP exerts on the spray drying process used in manufacturing washing powders is of great importance. The starting point for this process is an aqueous slurry in which a reaction between TPP and water is produced, thus giving rise to hexahydrated TPP (3). This hydration depends on several factors (4-7) and influences such characteristics as the consistency of the slurry, efficiency in production and detergent properties.

Moreover, hydration is one of the most significant stages in the process due to the fact that the slurry, before being injected into the spray drying tower, must possess specific and invariable properties. Therefore, the study of the rheological characteristics of a slurry is fundamental for operations like mixing, pumping and atomizing, which are part of the manufacturing process of laundry powders.

In a previous paper it was reported that, as the hydration of TPP takes place, a continuous increase in consistency, passing through a maximum when the reaction is completed, can be observed (7). These changes in consistency depend on the quality of TPP, temperature, concentration of slurry and concentration of hydrotrope. Subsequently, as is usual in concentrated suspensions (8), a stress overshoot due to the appearance of thixotropic phenomena may be detected. Furthermore, the degree of thixotropic effect increases with the concentration of nonaqueous substances in the slurry, the decrease of temperature, and the increase in the percentage of phase I in the TPP.

On the other hand, the rheological behavior of detergent slurries is difficult to predict because: (a) A high number of salts are present in the suspension; (b) The proportions of the soluble products can not be accurately defined; (c) Hydrates may be formed; (d) Thixotropic effects can appear, and (e) The slurry consistency is greatly increased after TPP is added. This exerts a negative influence on product handling and plant efficiency.

The purposes of the present paper are to analyze the rheological characteristics at steady shear of a detergent slurry containing TPP and to study the influence that some factors of technical interest exert on the rheological consistency of this slurry.

EXPERIMENTAL PROCEDURE

Equipment and method. The experimental results were achieved using a Brabender Plastograph, PL 3s model, which can be used in continuous processing operations and which provides the agitation necessary to obtain a well-integrated mixture of the components of the slurry. The equipment is fitted with a roller-type measuring head with two sigmoid, counter-rotating blades turning with different angular velocities (ratio of 3:2). A detailed description of this equipment and its operational conditions may be found in the bibliography on rheometry (9,10). The temperature is controlled by circulating water from a thermostat, which presents an accuracy of 0.2 ± 0.1°C, and using a thermocouple in direct contact with the sample.

Each sample was sheared at the maximum angular velocity up to steady state, and then torque measurements were taken. The angular velocity of the slower blade was modified from 180 to 20 rpm, which correspond approximately to shear rates between 300 and 25 s^{-1}.

Test variables. Due to the fact that rheological consistency depends on several factors, the following variables have been used:

- Quality of TPP: Three commercial products containing 94-96% TPP on the dry basis (56.5% P_2O_5), with different proportions of phase I and phase II TPP have been used: TPP-A (25% phase I); TPP-B (12% phase I); and TPP-C (1% phase I, partially hydrated).
- Concentration of slurry: A detergent formulation of

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standard composition was prepared by adding the following components to water in this order: sodium dodecylbenzene sulfonate, 6%; hydrotrope (sodium toluene sulfonate), 1%; fatty acid with a mol wt of 270.87 (mixture of palmitic and stearic acids) neutralized by sodium hydroxide, 3%; polyoxyethylene (EO 7) fatty alcohol, 3%; sodium silicate, 5%; anhydrous sodium sulfate, 51%; sodium carboxymethylcellulose, 1%, and TPP, 30%. The concentrations studied (C_w) were 65, 70, 75 y 80%, expressed as the weight concentration of all components, including TPP, and excluding only water.

- Temperature of the sample: Measurements have been carried out isothermally at 50, 55, 60, 70 and 80°C.
- Concentration of hydrotrope: The percentage of hydrotrope (sodium toluene sulfonate) was modified, thus the following values were used: 0, 1, 2, 5 and 10%.

RESULTS AND DISCUSSION

Rheological characterization. The Plastograph imparts a very complex shearing motion to the slurry, because of the irregular geometry of the blades and chamber. Consequently, the interpretation of rheological data obtained from the instrument is a very difficult and complicated process. Despite this difficulty, several approximate data treatment methods have been used to interpret the torque rheometer values (11-14), most of which were confirmed experimentally by the authors (15).

Therefore, an analogy between the sensor system of the Plastograph and two adjacent, concentric cylinder, rotational viscometers may be established. Thus, an equivalent roller radius was calculated with a margin of error lower than 4% (15). This radius allows calculations of shear stress, τ, and shear rate, $\dot{\gamma}$, from the experimental variations of the total torque, M, in function of the angular velocity of the slower roller, Ω_1. Some of the above mentioned variations may be observed in Figure 1, for a typical series of measurements.

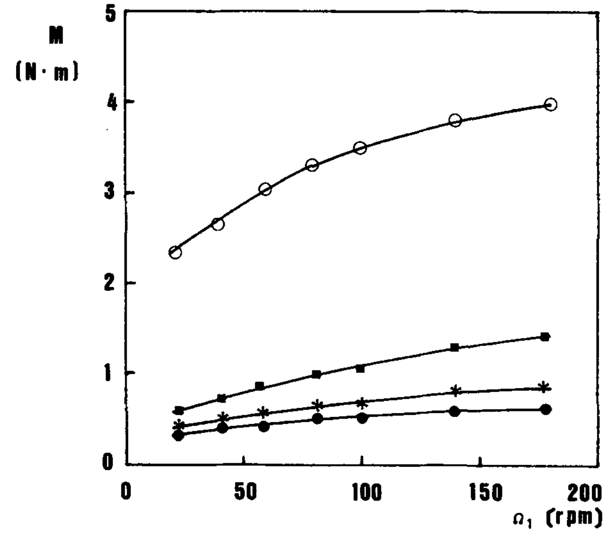

FIG. 1. Variation of the total torque, M, in function of the angular velocity of the slower roller, V_1, (C_w 65%, •; C_w 70%, *; C_w 75%, ■; C_w 80%, o).

The experimental results obtained in steady state fit, in most cases fairly well, the Casson and Herschel-Bulkley Models within the interval of shear rate studied:

$$\text{Casson: } \tau^{1/2} = \tau_0^{1/2} + (\mu_c\dot{\gamma})^{1/2} \qquad [1]$$

$$\text{Herschel-Bulkley: } \tau = \tau_{oh} + k'\dot{\gamma}^n \qquad [2]$$

where τ_0 and τ_{oh} are the yield values obtained by the Casson and Herschel-Bulkley Models, respectively; μ_c is the Casson plastic viscosity; and k' and n are the index of consistency and the flow index, according to the Herschel-Bulkley Model.

In order to calculate the rheological parameters for the Casson or Herschel-Bulkley Models from the Plastograph records, it is necessary, first of all, to fit the experimental data, M and Ω_1, to a power-law equation:

$$M = \alpha\, \Omega_1^b \qquad [3]$$

where α is a constant and b is equal to the flow index in the Ostwald-de Waele Model.

Because the radii ratio of the sensor system, a = R_b/R_c, has a value of 0.93, the Steiner approximation for the Casson Model may be used (16):

$$(1 + a)\tau_1^{1/2} = 2\tau_0^{1/2} + (1 + a)(\mu_c\dot{\gamma}_{N1})^{1/2} \qquad [4]$$

where τ_1 is the shear stress corresponding to the slower roller, which turns at an angular velocity of Ω_1, and $\dot{\gamma}_{N1}$ is the Newtonian shear rate for the roller 1.
Moreover, $\dot{\gamma}_{N1}$ is proportional to Ω_1:

$$\dot{\gamma}_{N1} = f\Omega_1 \qquad [5]$$

and the constant of proportionality, f, was found to be 14.97 when the equipment was calibrated previously (15).

The shear stress may be related to the torque value corresponding to roller 1 as follows:

$$\tau_1 = M_1/(2\pi h R_b^2) \qquad [6]$$

where h is the roller length and R_b the equivalent radius, which was also calculated by calibration (15).

Apart from that, the ratio for angular velocities is:

$$\Omega_2 = 1.5\Omega_1 \qquad [7]$$

and the pair of values M and Ω for both rollers follow a power-law equation:

$$M_1 = \beta\Omega_1^b \qquad [8]$$

$$M_2 = \beta(1.5\Omega_1)^b \qquad [9]$$

Thus, the experimental torque, M = $M_1 + M_2$, may be expressed as:

$$M = M_1(1.5^b + 1) \qquad [10]$$

Then equation [6] results in:

$$\tau_1 = M/[2\pi h R_b^2\,(1.5^b + 1)] \qquad [11]$$

The Steiner equation, [4], modified according to the characteristics of the torque-rheometer used, may now be written as follows:

$$M^{1/2} = 2Q(b)\tau o^{1/2} + (1 + a)Q(b)(f\mu_c)^{1/2}\Omega_1^{1/2} \qquad [12]$$

where the function Q(b) is expressed as:

$$Q(b) = [2\pi h R_b^2(1.5^b + 1)]^{1/2}/(1 + a) \qquad [13]$$

Consequently, Casson parameters τ_0 and μ_c may be calculated by correlating experimental data according to equation [12] when b has been obtained from the correlation of a power-law equation [3].

Both regressions may be considered satisfactory, especially the one corresponding to the Casson Model, as may be deduced from the values obtained for the residual variance VNJ (VNJ = 100 (1 − R^2) (N − 1)/(N − 2) where R is the linear regression coefficient and N is the number of pairs of experimental data.). For equation [3] the values of VNJ were lower than 5% in 90% of the cases. In the other cases, which correspond to the most extreme conditions of concentration and temperature, VNJ never exceeded a value of 8.4%. For equation [12], however, VNJ always remained below 3.7%; it presented values lower than 2.5% in 90% of the cases.

In reference to the rheological parameters in the Herschel-Bulkley Model, the procedure followed for calculation was different. The Krieger approximation as a solution of the Euler-McLaurin equation for a coaxial cylinder viscometer (17) has been applied:

$$\dot{\gamma}_1 = 2\Omega_1/[b(1 - a^{2/b})] \qquad [14]$$

This approximation is valid only if b is independent of angular velocity, as happened in all the systems studied. Furthermore, according to Darby, the above approximation provides a reasonably accurate evaluation of shear rate for materials with a yield stress under the majority of practical conditions encountered (18). This being the case, it is now possible to replace the shear stress in the Herschel-Bulkley Model applied to roller 1 with equation [11]. It is also possible to use τ_0, calculated from the Casson Model, instead of τ_{0h}, because it has been proved experimentally that they are not significantly different. Thus the result is the following equation:

$$M - Q(b)\tau_0 = KQ(b)\dot{\gamma}_1^n \qquad [15]$$

Consequently, parameters K and n may be calculated for each system by obtaining the value of b from the correlation of equation [3], the value of $\dot{\gamma}_1$ from equation [14] and by correlating M and $\dot{\gamma}_1$ according to equation [15].

The adjustment of the data to equation [15] is again satisfactory, because VNJ values are lower than 3.5% in 90% of the cases and never exceed 5.5%.

Once the calculation process was completed, the influence the variables described above exerted on the consistency of the slurry was analyzed on the basis of the following parameters: the yield value, calculated in accordance with the Casson Model (τ_0); the exponent of the Herschel-Bulkley Model (n), and the apparent viscosity at a shear rate of 200 s^{-1} (η_{200}) also calculated from the Casson Model. The variation of such parameters in function of the variables studied is represented in Figures 2, 3 and 4.

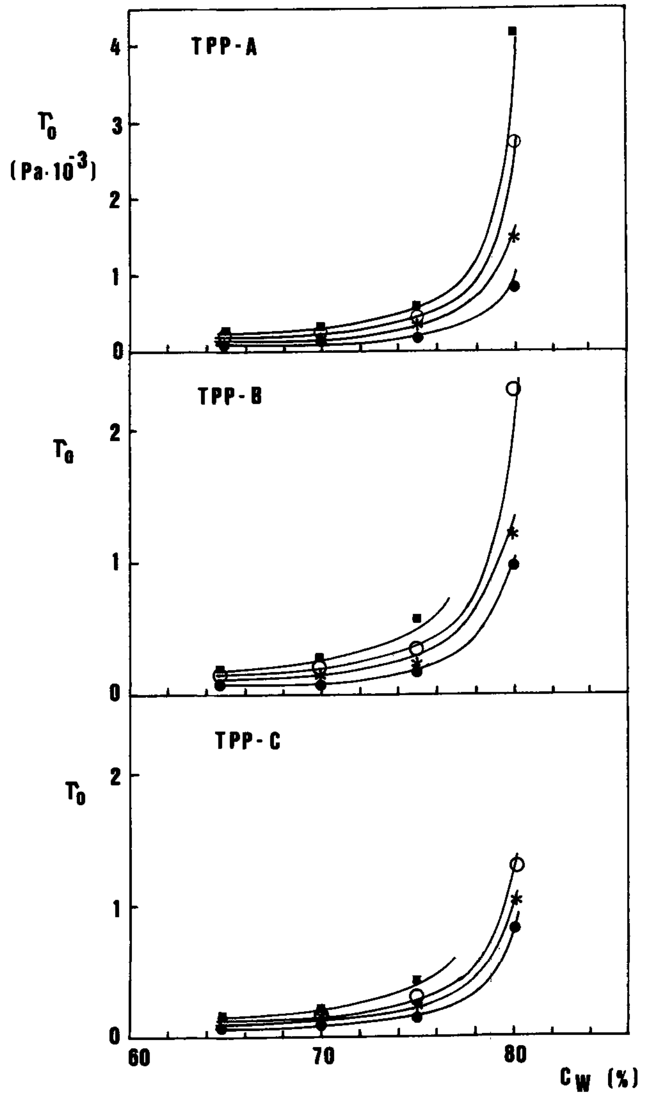

FIG. 2. Influence of temperature and concentration of dry substances on the yield value, τ_0. (C_w 65%, •; C_w 70%, *; C_w 75%, o; C_w 80%, ■).

A statistical study of the torque rheometer data at 60 and 140 rpm, M_{60} and M_{140}, respectively, and the above mentioned rheological parameters was carried out, using at least four measurements in each case. For these calculations a significance level of P = 0.05 was chosen. The results of this study are presented in Table 1 in terms of the average value, the confidence limits and the coefficient of variation of a series of measurements. This last statistic expressed as a percentage represents the ratio between the standard deviation of the sample and the average value.

Influence of quality of TPP. The phase I/phase II ratio in TPP has a certain dependence on the slurry consistency. Thus, values of the parameters τ_0, η_{50} and η_{200} increase by raising the phase I content, according to the following sequence of consistencies:

$$(\tau_0, \eta_{\dot{\gamma}})\text{TPP-A} > (\tau_0, \eta_{\dot{\gamma}})\text{TPP-B} \geq (\tau_0, \eta_{\dot{\gamma}})\text{TPP-C}$$

Although the experimental results follow this sequence, as is shown in Figures 2 and 4, the influence of the phase

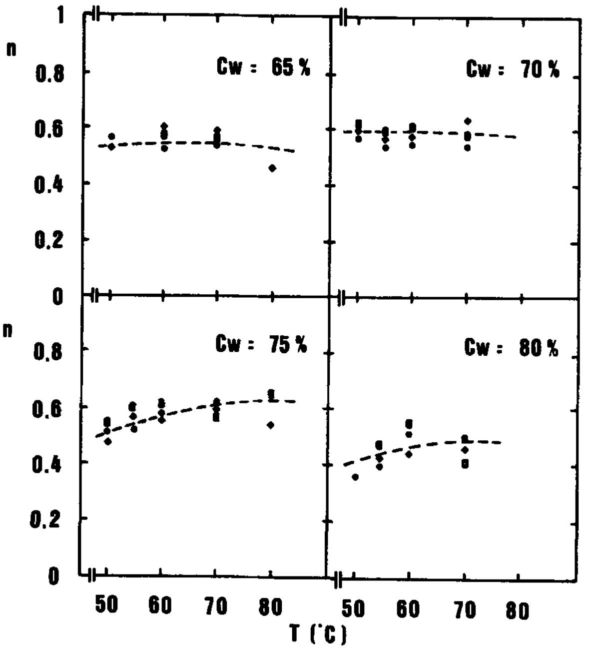

FIG. 3. Influence of temperature and concentration of dry substances on the exponent of the Herschel-Bulkley Model, n. (TPP-A, •; TPP-B, □; TPP-C, ■).

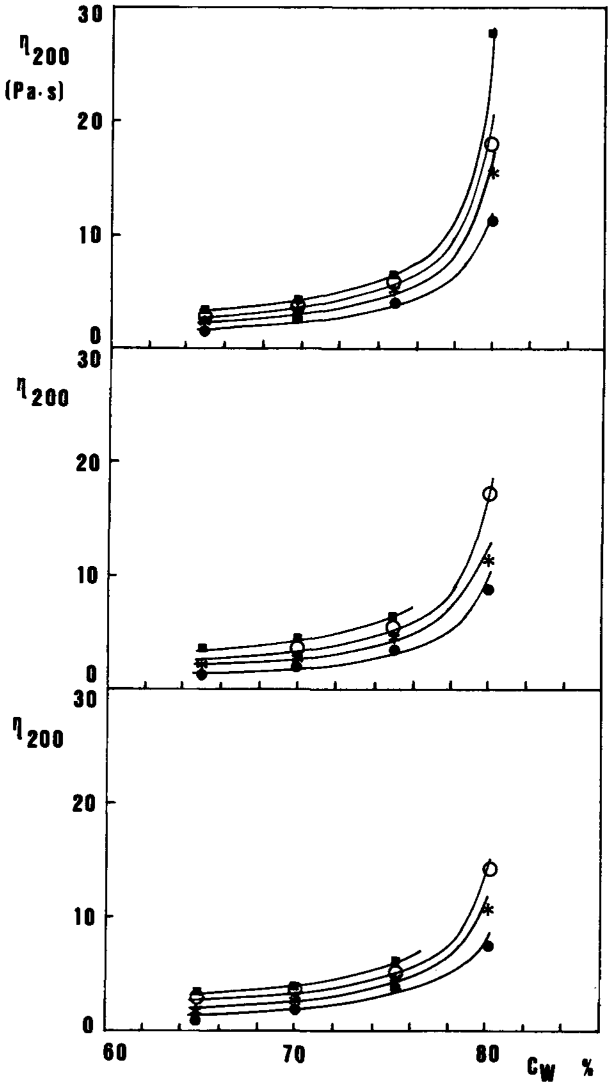

FIG. 4. Influence of temperature and concentration of dry substances on the apparent viscosity of the slurry at 200 s^{-1}, η_{200}. (C_w 65%, •; C_w 70%, *; C_w 75%, o; C_w 80%, ■).

I/phase II ratio on the rheological parameters of these slurries is not as significant as could be expected. This expectation is derived from the different characteristics that the hexahydrate crystals present for each TPP, because the velocity of TPP hydration depends upon the initial phase I/phase II ratio (4-7). Thus, phase I hydration occurs at a higher velocity than phase II, so that the hexahydrate crystals are smaller but more numerous in the first case (5), which results in a higher consistency.

The occasionally higher consistency of slurries containing TPP-B with respect to TPP-C may also be ascribed to the fact that this last variety of TPP was added already partially hydrated. The presence of these hexahydrate crystals aids the nucleation process and may provoke increases in growth rate eventually leading to higher sizes and, consequently, to thinner slurries (4, 5).

Influence of temperature and concentration of dry substances. An analysis of the dependence of the rheological parameters upon these variables shows that decreases in temperature (T) or increases in concentration of dry substances (C_w) provoke similar effects. This should not be surprising because both changes lead to a rise in the solid concentration of the slurry. Thus, as can be seen:

- The yield value increases when concentration is raised and temperature is diminished, as shown in Figure 2. Nevertheless, this effect is not significant at high values of temperature ($T > 65°C$) and at low values of concentration ($C_w < 70\%$).
- The dependence of apparent viscosity on shear rate, which is in relation to the flow index (n), becomes more important as the proportion of water drops (Fig. 3). Thus, a decrease in parameter n takes place by raising concentration or diminishing temperature. At low concentrations ($C_w \leq 70\%$), however, the flow index does not have any significant dependence on temperature.
- Apparent viscosity increases by raising the concentration of dry substances and diminishing temperature, but the first variable has a much more relevant influence.

The effect that temperature and concentration of dry substances exert on the rheological characteristics of the systems studied may be considered a result of the changes that both variables produce in the structure of these slurries.

Temperature may affect the consistency of a slurry because of the influence that it produces on the viscosity of the continuum phase, as well as on the degree of flocculation of particles (19). However, rheological parameters such as yield value (τ_0) or apparent viscosity ($\eta_{\dot\gamma}$) generally may be expressed as a function of temperature in an equation similar to the Arrhenius equation:

$$(\tau_0, \eta_{\dot\gamma}) = R\exp(s/T) \qquad [16]$$

where R and s are constants which may be determined

TABLE 1

Statistical Study of Torque-rheometer Data and Their Corresponding Rheological Parameters

		Average	Confidence limits	V(%)
TPP-A	M_{60}	564	± 22	3.12
T = 60°C	M_{140}	787	± 40	4.12
C_w = 70%	τ_0	203	± 12	4.59
	n	0.55	± 0.011	1.55
	η_{50}	7.75	± 0.23	2.38
	η_{200}	3.16	± 0.13	3.39
TPP-B	M_{60}	506	± 13	2.08
T = 60°C	M_{140}	757	± 29	3.02
C_w = 70%	τ_0	148	± 13	5.74
	n	0.58	± 0.015	2.68
	η_{50}	6.70	± 0.24	2.22
	η_{200}	2.98	± 0.09	1.86
TPP-C	M_{60}	473	± 33	5.70
T = 60°C	M_{140}	737	± 15	1.70
C_w = 70%	τ_0	118	± 18	9.45
	n	0.60	± 0.006	0.60
	η_{50}	6.06	± 0.24	4.01
	η_{200}	2.87	± 0.05	1.67
TPP-C	M_{60}	628	± 42	5.40
T = 70°C	M_{140}	981	± 55	4.53
C_w = 75%	τ_0	144	± 37	16.02
	n	0.62	± 0.023	2.30
	η_{50}	7.83	± 0.77	6.20
	η_{200}	3.80	± 0.22	3.60

experimentally and T is the absolute temperature.

The increase in the yield value, produced by a rise in concentration of dry substances, is a consequence of the increase in the solid characteristics of the slurry.

Some attempts at quantifying the relationship between yield stress (Bingham, Casson, etc.) and solids concentration may be found in the bibliography on concentrated suspensions. Although most of them are expressed as more or less complex functions of volume concentration (20-25), there are also exponential expressions which correlate yield value and weight concentration (26). Hence, an exponential function between the Casson yield stress and the weight concentration of dry substances may be obtained for these systems, as can be seen in Figure 2.

The dependence of apparent viscosity on concentration of dry substances of these slurries is similar to the one observed by other authors in disperse systems. While theoretical equations, such as Vand's (27), are suitable for diluted suspensions, empirical (28) or phenomenological equations (29,30) have to be developed for concentrated suspensions. Among these equations, a Mooney type expression (29), written in terms of weight concentration of dry substances, was used in this study:

$$(\tau_0, \eta_{\dot{\gamma}}) = R'\exp[s'C_w/(1 - C_w/C_{wm})] \qquad [17]$$

where R' and s' are constants and C_{wm}, according to the Mooney Model, should be the concentration of dry substances corresponding to the maximum packing volume fraction, at which τ_0 or $\eta_{\dot{\gamma}}$ tend to infinite values. Although C_{wm} is not independent of temperature, as the maximum volume fraction is, a unique value of C_{wm} has been used for the temperature range studied. The optimum adjustment of experimental data to equation [17] is obtained when C_{wm} has a value of 85%.

Moreover, the influence of both factors, temperature and concentration, may be combined to obtain the following equations:

$$\tau_0 = A\exp[a/T + bC_w/(85 - C_w)] \qquad [18]$$

$$\eta_{\dot{\gamma}} = B\exp[c/T + dC_w/(85 - C_w)] \qquad [19]$$

where A and B represent, respectively, the yield value and apparent viscosity at infinite dilution and temperature. Both of these parameters as well as parameters a, b, c, d and the multiple correlation coefficient, R, are presented in Table 2 for each variety of TPP.

TABLE 2

Values for Parameters in Equations (18) and (19)

		TPP-A	TPP-B	TPP-C
τ_0	R	0.976	0.961	0.978
	A	$1.90 \cdot 10^{-5}$	$6.69 \cdot 10^{-4}$	$9.33 \cdot 10^{-4}$
	a	5064	3793	3632
	b	0.196	0.197	0.192
η_{50}	R	0.989	0.986	0.980
	B	$2.99 \cdot 10^{-5}$	$5.22 \cdot 10^{-5}$	$1.92 \cdot 10^{-4}$
	c	3876	3659	3223
	d	0.168	0.161	0.149
η_{100}	R	0.991	0.987	0.977
	B	$5.57 \cdot 10^{-5}$	$3.79 \cdot 10^{-5}$	$1.76 \cdot 10^{-4}$
	c	3526	3629	3133
	d	0.160	0.150	0.138
η_{200}	R	0.990	0.981	0.971
	B	$1.31 \cdot 10^{-4}$	$2.96 \cdot 10^{-5}$	$1.70 \cdot 10^{-4}$
	c	3118	3598	3046
	d	0.150	0.138	0.125

Thus, for a given variety of TPP together with its corresponding experimental values, only one equation, [18] or [19], has been represented in Figures 2 or 3. Even though these correlations may be considered satisfactory, some deviations which take place because C_{wm} has a certain dependence on temperature can be observed at a concentration of 80%. At low temperatures the maximum packing fraction must correspond to a value of C_{wm} which is nearly 80%, and experimental data tend to an infinite value before the equation does. On the contrary, at high temperatures C_{wm} must be above 80%, going even higher than 85%, so that the tendency to infinite appears before the values predicted by the equation do. A correction of C_{wm} at each temperature undoubtedly would improve the above correlations, but it also would lead to unnecessary complications of the interpretation of results.

The modification of the parameters of equation [19] and consequently the fall in apparent viscosity with shear rate may be explained by assuming that a variation in the structure of the slurry is produced by shear forces (21-23, 31, 32). This structural change may lead to a reduction in the effective solid concentration as a result of the releasing of trapped water produced by the breakdown of particle aggregates as shear rate increases (24, 33).

Influence of concentration of hydrotrope. Regarding the influence of the concentration of hydrotrope, a decrease in both the τ_0 values and the slurry consistency may be noted (Fig. 5). This response may be attributed to the solubility of nonaqueous substances and the destruction of the structure of the system (34-36). Both effects have been explained by Friberg and co-workers (35,36), taking into account that hydrotrope molecules prevent the formation of liquid crystals by the long hydrophobic chains of surfactants, such as those present in these slurries. The short, bulky, aromatic compounds showing hydrotropic characteristics do not pack well in a liquid crystalline structure, so that the mutual stabilizing action of the hydrocarbon chains is lost, and instability takes place.

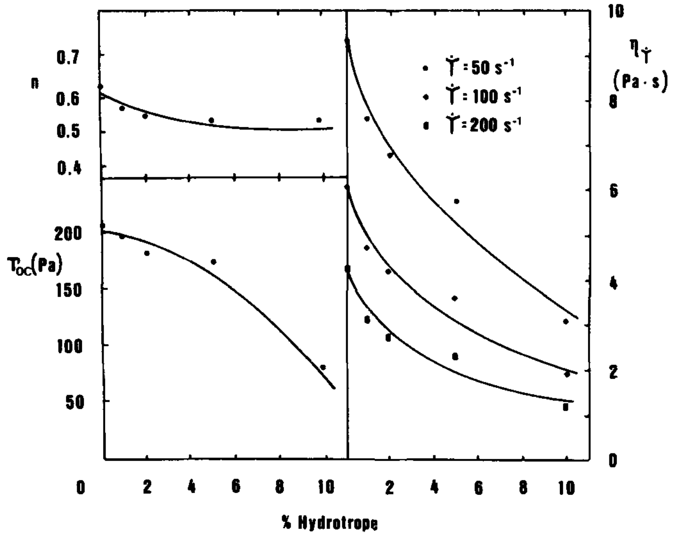

FIG. 5. Influence of hydrotrope content on the yield value, on the exponent of Herschel-Bulkley Model and on the apparent viscosity at 200 s^{-1}. (C_w, 70%; T, 60°C).

As a result, the aqueous micellar solubilization of low soluble substances, some of which are present in the formulations studied, is increased by the hydrotropic action.

Considering both of these effects, the structure that a slurry presents under shear and, consequently, its rheological consistency, may be directly related to the hydrotropic action. Nevertheless, in order to quantify the effect that the concentration of hydrotrope produces on the rheological parameters of these slurries, a more detailed study needs to be done. A study of such characteristics will be included in a later paper.

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