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INTERACTION OF WATER-SOLUBLE POLYMERS WITH CHRYSOTILE ASBESTOS, RHEOLOGICAL STUDIES

by

K. Park

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INTERACTION OF WATER-SOLUBLE POLYMERS WITH CHRYSOTILE ASBESTOS, RHEOLOGICAL STUDIES

SUMMARY

The interaction of chrysotile asbestos with a series of water-soluble polymers has been investigated using viscometry together with electrophoretic measurements.

Most water-soluble polymers studied interacted with chrysotile, altering the stability of the latter. The anionic polyelectrolytes exhibited the strongest interaction with chrysotile. A chrysotile suspension can be either dispersed or flocculated by the polyanions. Adsorption of anionic polyelectrolytes on chrysotile resulted in reversal of surface polarity of the latter from positive to negative. The adsorption was usually irreversible.

The non-ionic polymers also interacted with chrysotile to some extent. Some polymers either dispersed chrysotile or displayed protecting action for chrysotile in the presence of anions which normally flocculate chrysotile, but their interaction capacity was much less pronounced compared to that observed with the anionic polymers.

A typical cationic polyelectrolyte behaved very differently from both the anionic or non-ionic polymers. For instance, no dispersion was observed in a chrysotile suspension to which the cationic polyelectrolyte had been added. It appears that cationic polyelectrolytes in general contribute to increase in viscosity of the suspension containing chrysotile and the polymer.

Mechanisms of interaction between polymer molecules and chrysotile particles have been discussed.

INTRODUCTION

It has been known that a small amount of lyophilic colloid* can sensitize or flocculate lyophobic colloids.* The surface of lyophobic particles can be completely covered by the lyophilic colloid (protective action), thus altering the interfacial properties and hence the stability of the original system. It is generally assumed that the sensitization toward flocculation is due to the formation of agglomerates of the lyophobic particles held together by the lyophilic substance. The protecting action against flocculation is a result of complete coverage of the lyophobic particles by lyophilic colloids.[2,3]

* The lyophobic colloid is essentially the two-phase system with a large total interfacial area, whose stability property primarily depends on pH or electrolyte concentrations. On the other hand, "lyophilic" colloids can be considered as a macromolecular solution whose stability is mainly governed by the degree of solvation.[1]

As are most inorganic colloids, an aqueous dispersion of chrysotile asbestos behaves as a lyophobic colloid. The particles of chrysotile asbestos are highly asymmetric so their frequency of collision is large. Since they possess electrical double layers, the stability of their suspensions depends greatly on pH or electrolyte concentration. Flocculation, occurring in alkaline solutions or in the presence of dissolved salts, causes formation of a structure consisting of loosely or strongly bonded aggregates. On the other hand, a stable sol of chrysotile can be obtained by a simple pH adjustment with acetic acid.[4] The chrysotile suspension is capable of interacting with various "lyophilic" colloids,* also changing its stability properties, i.e., the asbestos can be either flocculated or deflocculated by interacting with the "lyophilic" colloids. Study of the mechanism of such an interaction is the subject of this report.

The interaction properties have been deduced mainly from viscometric and electrophoretic measurements.

EXPERIMENTAL

The water-soluble polymers used in the present investigation were commercial products. Their trade names, manufacturers, chemical names, etc. are recorded in the Appendix.

Aqueous solutions of the high molecular weight substances were prepared by the procedures suggested by the manufacturers.

Aqueous suspensions of asbestos were prepared by mixing in a Waring Blendor an appropriate amount of high-purity opened asbestos with distilled water. Dispersed suspensions were obtained by adjusting the pH to 4.5 with acetic acid.

The chrysotile suspensions containing various amounts of polymer were prepared by adding the desired amount of polymer solution drop by drop to the asbestos suspension while it was being stirred (stirring motor).

After adding the high molecular weight substances, the suspension was rested for 12 or 24 hours before viscosity or polarity measurements were made. It is known that adsorption of macromolecules onto solid surfaces from solution is normally a slow process, so the equilibrium is reached in much longer time than in adsorption of small molecules.[5] It was found that an aging period of 12 to 24 hours was usually satisfactory to obtain reproducible results. In order to eliminate any variation that may result from the preparation of different asbestos suspensions, one stock suspension was used for each series of experiments.

The instrument used for measuring the flow properties was a Fann-35 rotational viscometer (a Couette type). The nominal shear rate** range used was 2.3 to 1022 sec.$^{-1}$.

* Solutions of water-soluble polymers are typical lyophilic colloids.

** When the flow properties of aqueous chrysotile suspension are measured in a concentric cylinder viscometer, it is often necessary to take into account the variation of apparent viscosity with the bob-cup diameter ratio. This occurs because of the pronounced non-Newtonian characteristics of a chrysotile suspension, and so the true shear rate could be significantly different from the apparent shear rate which is generally calculated from a Newtonian viscosity equation. Therefore, an appropriate correction must be made if the flow data for a chrysotile suspension are to be accurately represented.

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The polarity of the chrysotile surface charge after the polymer treatment was determined by either microelectrophoresis or electroosmosis. The electrophoresis apparatus (Zetameter) was used when a suspension was sufficiently dispersed. On the other hand, the electroosmosis apparatus was used for flocculated systems.

The flocculation test of a suspension of polymer-treated asbestos was made by the following procedure: In a series of graduated cylinders, an increasing amount of electrolyte solution was added to each measured portion of dilute suspension containing asbestos and polymer, and measurements of the flocculated volumes were made 1 to 20 hours after preparation. The filtration rates of the suspensions of a polymer treated asbestos were measured with a Baroid filter press.

RESULTS AND DISCUSSION

I. Anionic Polyelectrolytes

A. Flocculation-Deflocculation of Chrysotile by Anionic Polyelectrolytes

Stabilization or flocculation of inorganic dispersions by adsorbed anionic polyelectrolytes is a recognized phenomenon, and a number of experimental works on the subject have been reported.[6-9] However, the investigations thus far reported are mostly involved with interaction between anionic polyelectrolytes and solid particles of the same charge, such as kaolinite[7] and anatase.[8] Studies on adsorption of anionic polymers on surfaces of positive charges have been rarely found in the literature.

Considering the fact that the particles of chrysotile asbestos bear electropositive surface charge in aqueous media,[4] they would be expected to exert a strong attraction for the anionic polyelectrolytes. It has been found indeed that the anionic polyelectrolytes are very specific in altering the surface property of chrysotile asbestos.

Addition of a small amount of an anionic polyelectrolyte to a flocculated suspension of asbestos causes the latter to disperse, and the degree of dispersion increases to a certain limit with the amount of polymer added. Figure 1 shows the variation in the viscosity of a 0.5% (by weight of total suspension) asbestos suspension with the amount of CMC added. CMC is a commerical name of sodium salt of carboxymethyl cellulose. Effects of three synthetic linear polyelectrolytes, EMA-11, Gantrez An-169, and Gantrez An-4151, on the flow property of chrysotile suspension are also shown in Figure 2.

It is seen (Figures 1 and 2) that the viscosity of 0.5% chrysotile suspension is markedly reduced in the range, 1 to 5 pha (parts per hundred of asbestos) of polymer addition. In general, the decrease in viscosity is followed by a gradual increase at high polymer concentrations. The 0.5% chrysotile suspension has non-Newtonian (either plastic of pseudoplastic) flow characteristics, whether or not is is treated with polymer. Accordingly, its viscosity value can be specified only for a certain shear rate. It has been found that comparison of "apparent viscosity" is generally valid if it is used only as a measure of dispersion or flocculation.

The dispersing action of anionic polyelectrolytes is also illustrated by the data in Figure 3. There it is seen that the filtration rate of an asbestos suspension is drastically reduced on addition of CMC, indicating that there is a close correlation with the results of flow measurement (cf. Figure 1).

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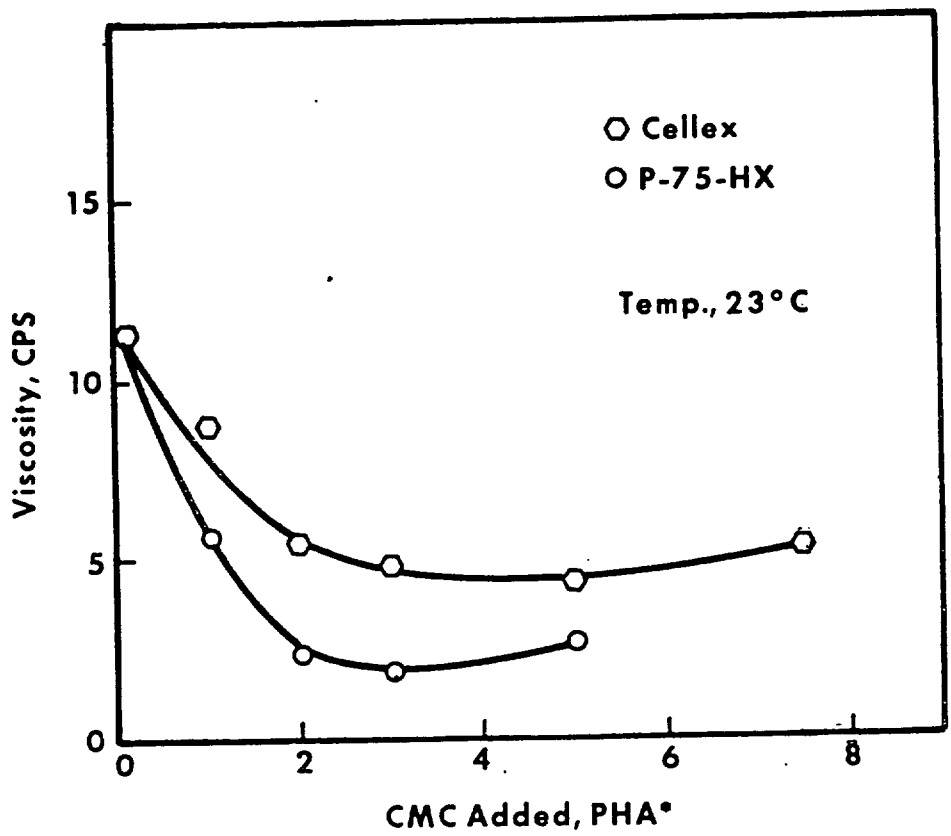


FIG 1 VISCOSITY VARIATION OF ASBESTOS SUSPENSION WITH CMC CONCENTRATION. SUSP., 0.5% BY WT. SHEAR RATE, 1000 SEC^{-1}

(* PARTS PER HUNDRED OF ASBESTOS)

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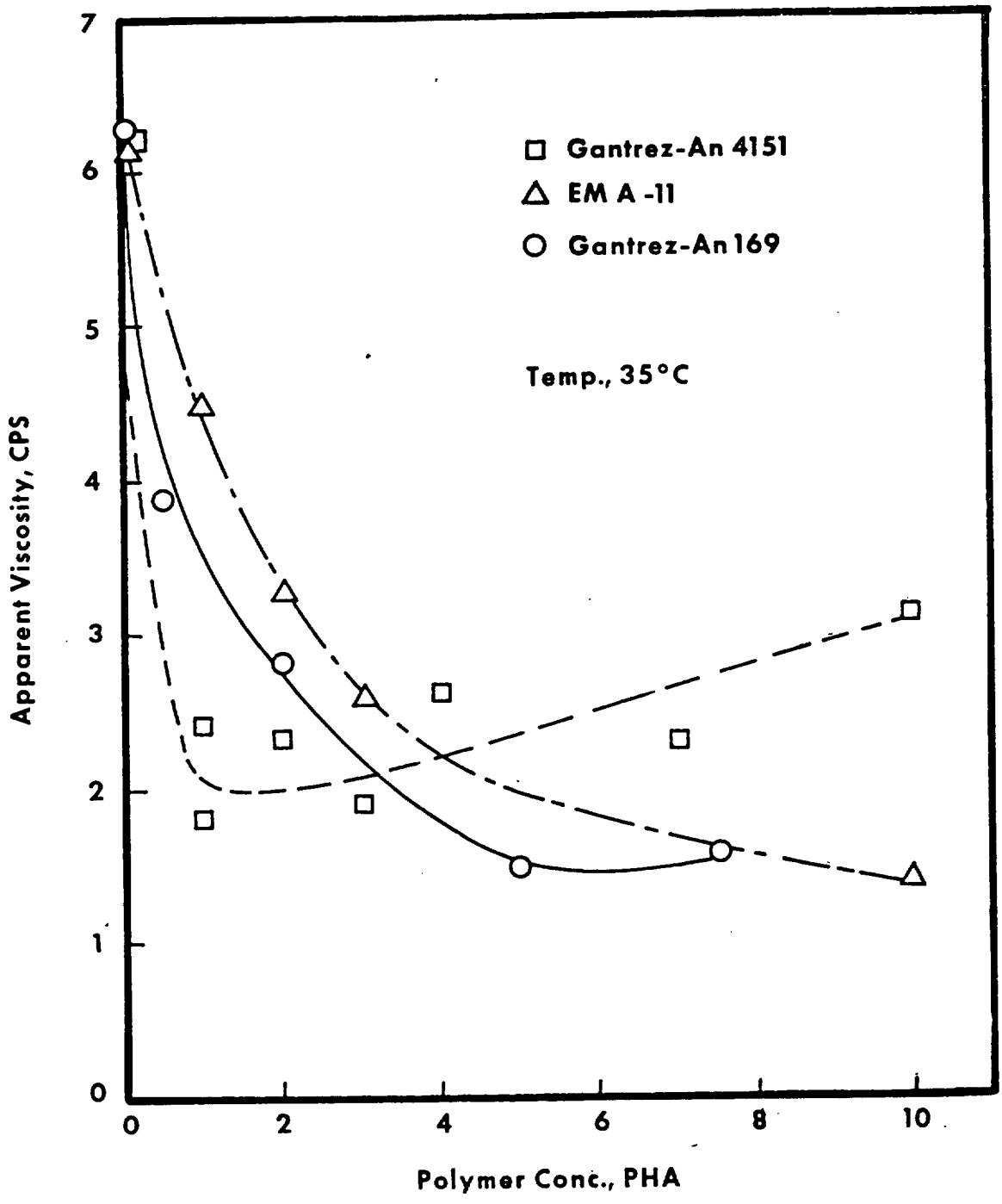


FIG. 2 EFFECT OF THREE DIFFERENT ANIONIC POLYELECTROLYTES ON APPARENT VISCOSITY (SHEAR RATE = 511 SEC.$^{-1}$) OF 0.5 WT. % ASBESTOS SUSPENSION. PH of the Original Suspension $\simeq$ 9

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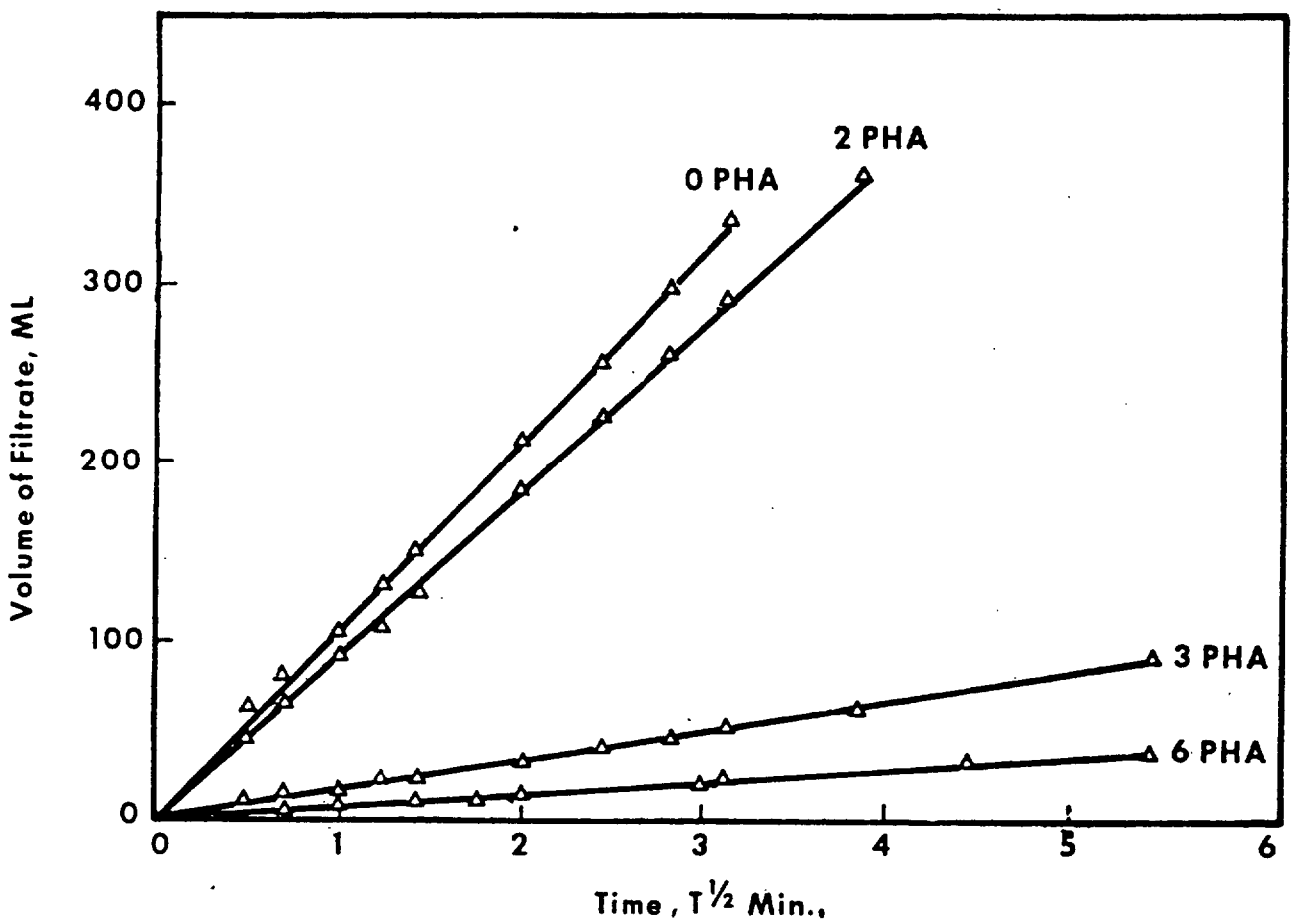


FIG 3 FILTRATION RATE OF ASBESTOS SUSPENSIONS AT DIFFERENT CMC (CELLEX) CONCENTRATIONS (PHA: PARTS PER HUNDRED OF ASBESTOS)
SUSP., 2% BY WT. TEMP., 23°C PRESS., 50 PSI

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The fact that viscosity as well as filtration rate decrease on addition of anionic polyelectrolytes clearly demonstrates that the chrysotile particles are dispersed, i.e., the aggregates of chrysotile fibers are separated into smaller units in the presence of the macromolecular substances. There is little doubt, as it will become more evident in a later discussion, that the dispersion of chrysotile suspension occurs because the chrysotile particles are covered by the polyelectrolyte molecules which repel one another because of their ionic charges on the chain.

It is interesting to note that the filtration rate decreases when the polymer addition is increased above 3 pha, whereas viscosity begins to increase at the same polymer addition. This implies that the largest dispersion does not necessarily occur between 2 to 4 pha of polymer addition. In fact, degree of dispersion was greater at 6 pha than at 3 pha, judging by the visual observation.

The increase in viscosity at higher level of polymer addition seems to be due to the increase in viscosity of the dispersing medium (polymer solution). The CMC molecules which are not attached to the chrysotile particles should act as independent kinetic units; thus, they should contribute to the increase in viscosity of the system.

The anionic polyelectrolytes are also capable of flocculating chrysotile asbestos. This flocculating effect seems to conform to the general behavior of lyophobic colloids, which is called "sensitization"[2] or "adsorption coagulation."[10]

The viscosity variation of 1% chrysotile suspension with addition of an anionic polyelectrolyte is shown in Figure 4. The Carbopol is believed to be a poly (acrylic acid) of extremely high molecular weight derived from the use of a certain cross-linking agent.[11] It is seen that the cross-linked polyelectrolyte behaved very differently compared to the previously mentioned linear polyelectrolytes. Addition of a small amount of Carbopol-941 resulted in extensive flocculation of chrysotile, as indicated by the sharp increase in viscosity at 0.5 pha of polymer addition (Figure 4). The flocculation was so extensive that a gentle stirring gave rise to a large single lump of chrysotile separated from suspending medium. However, the lump was readily broken into smaller ones by a vigorous agitation.

It appears that the strong flocculating power of Carbopol, compared to other anionic polymers, is due to its exceptionally large molecular size, which favors particle-to-particle bridging. It is reasonable that large molecules, because of their bulkiness, more readily interfere with the chrysotile aggregates, compared to small molecules; thus, they may be partially adsorbed to one chrysotile particle, with the remaining portion being extended to other particles.

It is seen in Figure 4 that the initial increase of the apparent viscosity on addition of a small amount of Carbopol was followed by a decrease when the polymer concentration was increased. The decrease in viscosity was again followed by a subsequent increase. The data also show that the variation of viscosity was much less pronounced at a high shear rate than at a low shear rate. It was observed that the shift from the upper curve (viscosity values at the higher shear rate) to the lower one (viscosity values at the lower shear rate) was almost irreversible, i.e., the shearing gave a permanent disruption of aggregated structure of chrysotile particles.

The fact that there exists only a weak bonding force between the macromolecules and the chrysotile aggregates suggests that adsorption of Carbopol molecules does not take place at a large enough number of sites to give a strong bonding.

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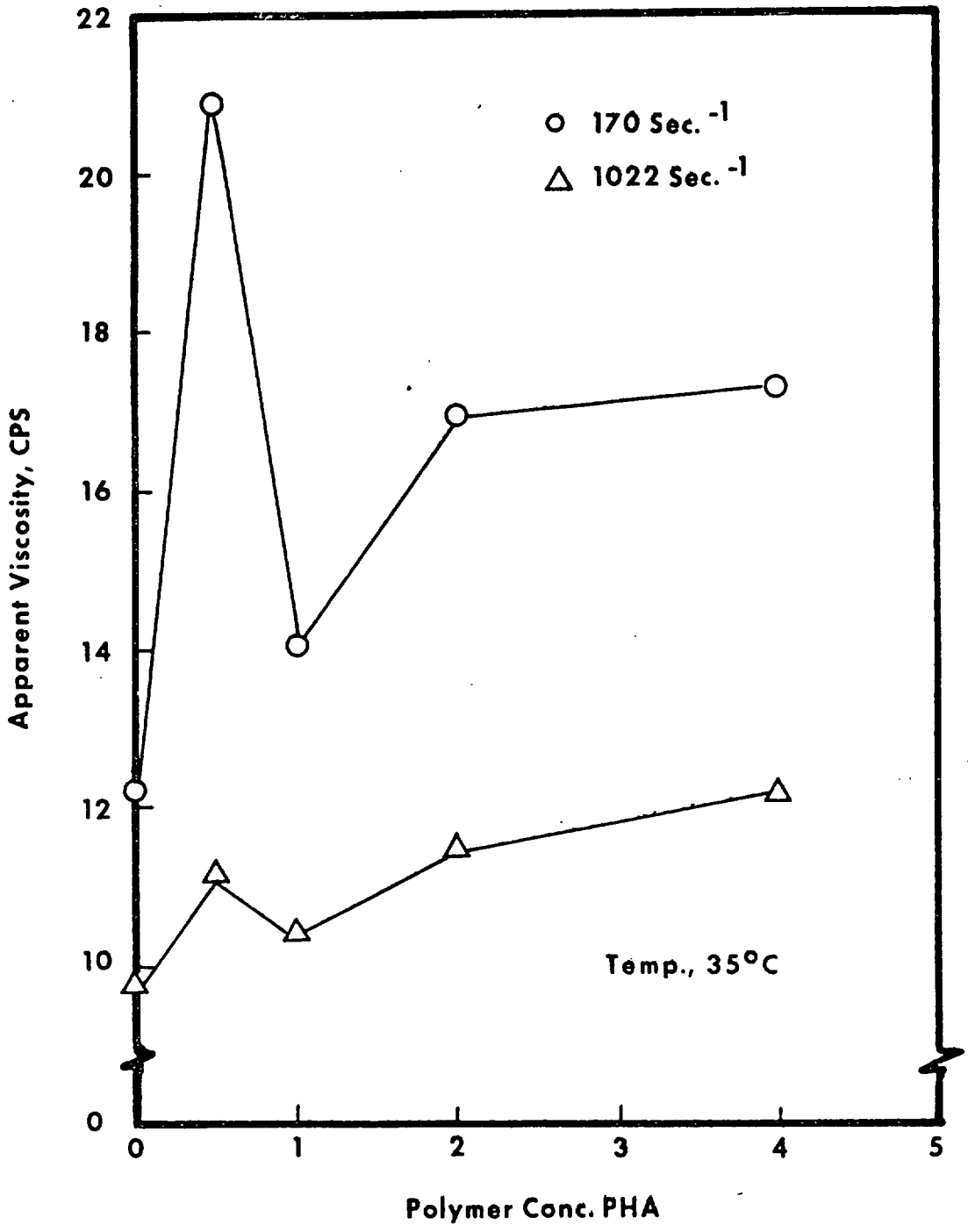


FIG. 4 VISCOSITY VARIATION OF 1.0 WT. % ASBESTOS SUSPENSION WITH CONCENTRATION OF CARBOPOL-941 (A CROSS-LINKED ANIONIC POLYELECTROLYTE). The Data Represent Viscosity Values at Two Different Shear Rates.

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The linear polyelectrolytes can also flocculate a chrysotile asbestos when the polymer is added to a sufficiently dispersed suspension (e.g., a suspension of pH ≈4.5). Figure 5 represents the effect of EMA-11, a typical linear anionic polyelectrolyte, on the apparent viscosity of a chrysotile suspension (pH ≈4.5). The apparent viscosity is sharply increased by addition of a small amount of EMA, followed by a maximum, then is decreased to a plateau value. Although the increase in viscosity beyond the plateau was not observed in the range of polymer addition, it would be expected that the viscosity will eventually increase if the polymer addition is further increased, i.e., above 8 pha.

In Figure 6, a diagramatic scheme is shown to illustrate the effect of polymer addition on flocculation-deflocculation of chrysotile asbestos. Figure 6 (1) is represented as a dispersed state of chrysotile suspension and the subsequent figures, (2), (3), and (4), illustrate the effect of polymer addition.

A small amount of polyelectrolyte molecules flocculates chrysotile extensively (Figure 6 (2)). This gives rise to a sharp increase in viscosity as is seen in Figures 4 and 5. Based on the established concept of flocculation of lyophobic colloids, it is reasonable to assume that there are two factors which may be responsible for the flocculation of chrysotile in the presence of a small amount of polymer. The first, of course, is the previously mentioned bridging-mechanism. The second possible mechanism would be the flocculation which results from the reduction of surface potential of the chrysotile. The flocculation could then occur, as a result of depression of electropositive potential of chrysotile, through the dominance of van der Waals type forces between the chrysotile particles, causing them to adhere to each other. Although the latter type of flocculation is a possiblility, it is generally recognized that the former is a major mechanism in the flocculation of lyophobic particles by macromolecules.[9,12] In any instance, it would be inferred that the second type of flocculation would be less probable if either the molecules are too large, as would be the case with Carbopol, or the adsorption takes place in a thick multilayer.*

As the polymer addition is further increased, the entire surface of chrysotile particles may be covered with the polyelectrolyte molecules, and the chrysotile particles which overcome the bridging force of molecules will stay separate from each other as a result of the repulsion of the ionic charges of the adsorbed molecules (Figure 6 (3)). The dispersion thus effected should give rise to decrease in viscosity, as seen in Figures 4 and 5, where viscosity begins to decrease with polymer addition beyond the maximum.

If the amount of polymer added is more than what is needed for a complete coverage of chrysotile particles, the macromolecules which are not bound to the chrysotile particles will function as independent flow units (Figure 6 (4)), causing an increase in viscosity. This seems to be an obvious explanation for the observed increase in viscosity at a higher level of polymer addition (Figures 1, 2, and 4).

B. The Reversal of Surface Polarity of Chrysotile

The addition of anionic polyelectrolytes to a chrysotile suspension often results in reversal of surface charge of the chrysotile particles from positive to negative. The adsorption is usually irreversible. In typical tests used to study

* van der Waals attraction normally becomes very weak beyond c. 100 Å of particle-to-particle distance.[13]

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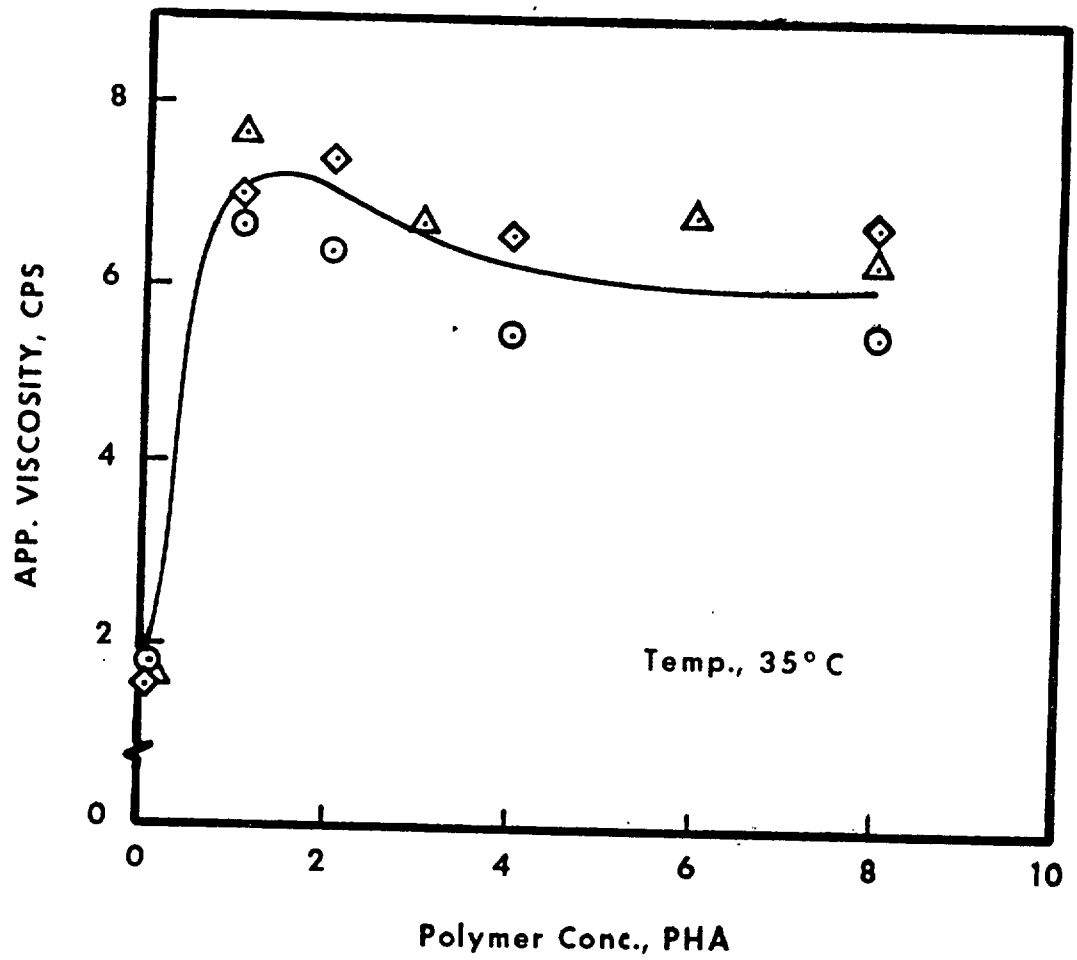


FIG. 5 VARIATION IN APPARENT VISCOSITY (SHEAR RATE = 511 SEC.$^{-1}$) OF 1 WT. % ASBESTOS SUSPENSION (PH = 4.5) WITH EMA–11 ADDITION.

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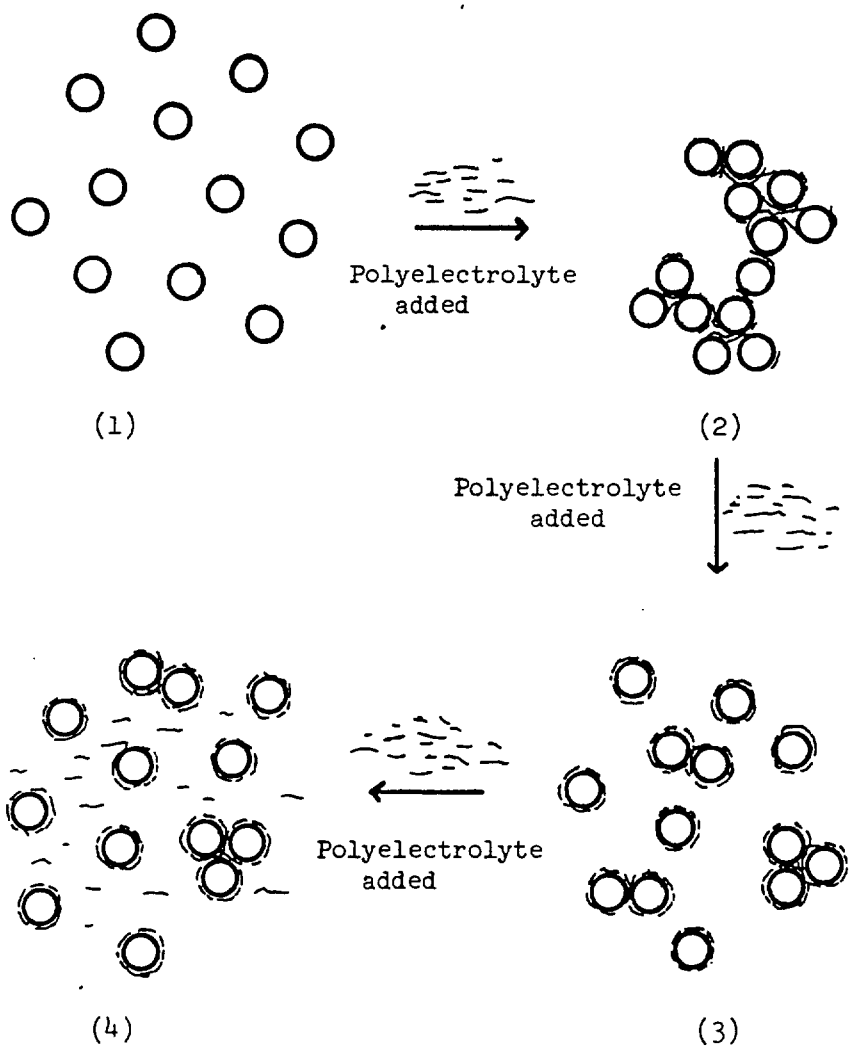


FIGURE 6 - Diagram illustrating a mechanism of Flocculation-deflocculation of chrysotile by anionic polyelectrolytes. Chrysotile fibers are represented by circles, which may be viewed as their cross sections (cf. Figures 4 and 5).

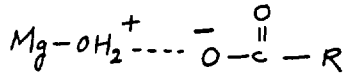
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this effect, a polymer-treated asbestos suspension was filtered and the filter cake was dried. Then it was redispersed in a large amount of pure water to reduce the solution concentration of polyelectrolytes. Whether the polarity of surface charge remained negative after redispersion was used as a qualitative criteria of irreversibility of surface polarity.

Table I summarizes the polarity of surface charge of chrysotile treated with four different anionic polyelectrolytes. It is seen that the polarity change can be reversible unless the polymer concentration is sufficiently high (e.g., EMA-11 and Gantrez-An 4151). The data show that the minimum concentration of each polyelectrolyte at which the irreversible polarity-change occurs ranges from 2 to 4 pha.

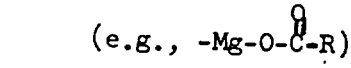
In view of the general concept of adsorption of macromolecules on solid surfaces,[12] it is rather obvious that the strong adsorption is due to the multiple bonding between the carboxyl groups on polyelectrolyte chains and the chrysotile surfaces.

The present investigation by no means provides information on the true nature of bonding between the polyelectrolyte molecules and the chrysotile surfaces. Nevertheless, it may be reasonably suggested that the bonding is electrostatic in nature, i.e., the cationic chrysotile surfaces form an electrostatic bond with the negatively charged carboxyl groups of the polymer chain, which may be depicted as follows:



where Mg represents the octahedrally coordinated magnesium atom in the crystal structure of chrysotile, and R represents the polymer chain.

In addition to the electrostatic attraction between the dissimilar charges of polymer molecules and chrysotile, it seems probable that the adsorption is assisted by van der Waals forces. There are a number of other, less likely but possible, bonding schemes, such as ion exchange (removal of physically adsorbed hydroxyls on chrysotile surfaces by the ionized carboxyl groups of the polyelectrolytes) or formation of a surface compound



Nevertheless, the ultimate clarification of the bonding mechanism must come from quantitative adsorption studies.

It is interesting to estimate how much polyelectrolyte would be required for a complete coverage of chrysotile fiber surface and how it compares with the minimum amount of polymer needed for the polarity-reversal.

The calculation was made for a hypothetical system of chrysotile asbestos covered in monolayer by linear acrylic acid whose average molecular weight is 10,000. The area occupied by a monomeric unit of polyacrylic acid was determined[14] to be 22 Å. Using this value, and assuming that the polymer molecules lie flat on the surface of chrysotile, the minimum amount of the polymer for covering the surface of chrysotile was calculated as follows:

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TABLE I

Polarity of Surface Charge of Chrysotile Asbestos
Treated with Four Different Anionic Polyelectrolytes

(The data show the minimum concentration of each polymer, at which the irreversible polarity-reversal occurs.)

Polymer	Amount of Polymer Added (pha)	pH of Suspension	Polarity	
			Original Susp.	Redispersed*
Cellocize (P-75-HX)	1.5	-	positive	-
	2.0	9.1	negative	negative
Gantrez-An 169	1.1	-	positive	-
	2.1	7.5	negative	negative
EMA-11	0.94	-	positive	
	1.4	-	negative	positive
	1.9	8.1	negative	negative
Gantrez-An 4151	0.91	-	positive	
	1.8	-	negative	positive
	3.6	8.9	negative	negative

* The suspension treated with polymer was filtered and the filter cake was dried. Then it was redispersed in distilled water.

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Taking the surface area of chrysotile[4] as 60 $m.^2/g.$, the number of molecules needed for covering 1 gram of chrysotile, is 1.96×10^{18} (3.3×10^{-6} moles). From these values, it was found that approximately 0.03 gram of polymer (i.e., 3 pha of polymer) covers the entire surface of 1 gram of liberated chrysotile asbestos fibers.

Assuming 2 pha as the minimum polymer concentration at which the surface polarity of chrysotile reverses from positive to negative (cf. Table I), the number of polyacrylic acid molecules adsorbed by a single chrysotile fiber to give the polarity reversal was calculated to be 18,000. In this calculation, the number of individual fibers which one gram of chrysotile contains,[15] was assumed to be 10^{14}. Incidentally, the chain length of the hypothetical polyacrylic acid molecule, in its fully extended conformation, was calculated to be 353 A, which is comparable to the average diameter of a single chrysotile fiber (300 A).

C. Effect of Simple Inorganic Electrolytes on the Stability of Polymer-Treated Asbestos

The results from the flocculation tests (Table II) illustrate the effect of sulfate ions on the stability of a chrysotile suspension. It is seen that the EMA treated suspension is stable in the presence of sulfate ions, i.e., no flocculation was observed in the EMA-asbestos suspension, but the untreated suspension flocculated strongly on addition of sulfate ions.

This seems to prove that the electropositive surface of chrysotile is completely enveloped by the polyelectrolyte molecules whose stability is not sensitive toward sulfate ions. In other words, the anionic polyelectrolytes function as protective colloids for chrysotile in the presence of sulfate ions (or, anionic species in general).

The effect of cations of different valency types and of pH on the stability of chrysotile suspension treated with CMC is summarized in Table III. Flocculation of the treated suspension by the electrolytes appears to follow the Schulze-Hardy rule, i.e., the flocculating power of cations for the negatively charged suspension of CMC-Asbestos increases as the valence of the ions becomes higher. The stability of the suspension was essentially unaffected by the monovalent cations, but was greatly affected by the trivalent ions. The divalent cations showed an intermediate effect. However, the salt-resistant CMC (Cellocize, P-75-HB)[11] showed considerably higher stability in the chrysotile suspension than did the other CMC form, i.e., Cellex*. Discussion about the dependence of salt sensitivity of CMC on the degree and the uniformity of carboxylic substitution is given elsewhere.[17]

The suspension of CMC-treated asbestos was not susceptible to flocculation with pH variation, e.g., it showed stability in the pH range, 4 to 10. Since the free acid form of CMC is insoluble in water,[17] it is expected that the CMC-treated asbestos will be flocculated if pH is further lowered, e.g., below pH ≈2.

* No comparative study of different forms of Cellocize was made. Zimmerman and Stratta[16] reported that at lower resin concentration there was no appreciable difference in salt sensitivity between the ordinary H-type and the HB-type Cellocize.

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TABLE II

The Results of Flocculation Test Illustrating the Protective Action for Chrysotile in the Presence of Sulfate Ions

Dispersoid conc. - 0.2 wt. %
Amount of polymers added - 2% by weight of asbestos

Suspension	Amount of K_2SO_4 added, Meq./l 0.02	0.1	0.3
Orig. susp. without polymer (pH = 4.5)	VF	VF	VF
EMA-11 Added	S	S	S
Gantrez-M 154 Added	SF	VF	VF

TABLE III

Effect of Electrolytes on the Stability of c. 0.1% Asbestos Suspension Treated with Two Different CMC

Amount of polymer added ≈ 4 pha
Set period ≈ 18 hr.

(1) Baroid CMC (Cellex)

Salt	Amount of Salt Added, Meq./l 0.005	0.05	1	2	5
KCl	-	S	S	S	S
$BaCl_2$	-	S	S	SF	F
$AlCl_3$	F	VF	VF	VF	VF

(2) Cellocize, P-75-HB

Salt	Amount of Salt Added, Meq./l 0.005	0.5	2	5
$BaCl_2$	S	S	S	SF
$AlCl_3$	F	VF	-	-

(3) The pH Effect

The suspension of CMC-treated asbestos was stable in a wide range of pH. For instance, no flocculation was ever observed when pH was varied from 4 to 10.

Descriptions: Stable (S) Flocculated (F)
Slightly flocculated (SF) Very flocculated (VF)

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D. Thickening of a Polyelectrolyte Solution by Chrysotile

Figure 7 shows typical flow curves of chrysotile suspensions in 0.5 weight per cent CMC solution. These are the equilibrium down-curves,* obtained after prolonged shearing. In order to minimize climbing of the fluid along the bobbin, known as the Weissenberg effect, the flow data were obtained in a relatively low shear rate region. Judging from the relatively large shear stress values at a low shear rate range, the suspensions of 1% (by total weight of suspension) or higher of dispersoid concentrations appear to have yield stresses. Above 2% of dispersoid concentration, the suspensions displayed an excellent stability, showing no phase separation when they were allowed to set for several months.

In Figure 7, it is shown that the consistency of the CMC solution decreases on introduction of 0.2% chrysotile (by weight of total suspension), then increases as the dispersoid concentration is further increased. The increase in consistency on addition of 0.5% asbestos is much less pronounced in this system, compared to the chrysotile suspension in other media, e.g., pure water[4] or Polyox solution (cf. Figure 13). The effect of addition of chrysotile on the relative apparent viscosity of 0.5% CMC solution is shown in Figure 8. The viscosity values were calculated from the data in Figure 7. Here, it is shown that the apparent viscosity of the 0.2% chrysotile suspension is smaller than that of the suspending medium.

The decrease in viscosity on introduction of insoluble solid particles into a liquid cannot be explained by usual hydrodynamic considerations. However, in view of the fact that the polyelectrolyte molecules are adsorbed by chrysotile, it seems probable that the decrease in viscosity occurs because the solid particles remove the macromolecules from the solution phase, reducing the total number of flow units in the system. In other words, there are two diverging effects on viscosity in the system; adsorption of CMC molecules tends to decrease viscosity, but the increase in hydrodynamic volume of chrysotile particles contributes to the increase in viscosity. The former effect is dominant in a lower concentration level of chrysotile, but is overwhelmed by the latter as the asbestos content increases.

Curve "b" in Figure 9 shows typical thixotropic hysteresis obtained from a 3% (by weight of total suspension) chrysotile suspension in 0.5% polymer solution. The upward arrow points to the up-curve, which was obtained by increasing the shear rate. On the other hand, the downward arrow indicates the down-curve, which was obtained while the shear rate was decreased from 113 $sec.^{-1}$ to 2.3^{-1}. The CMC (Cellocize, P-75-HX) solution itself (Curve "a") is pseudoplastic and shows no thixotropic hysteresis as was also observed by Stratta.[11]

Evidently, the observed stability of chrysotile dispersion in CMC solution is associated with the thixotropic structures formed as a result of interaction between the CMC molecules and chrysotile particles. The spatial network formed by the dispersed chrysotile particles should prevent the suspension from settling, which is often recognized as a characteristic of an unstable suspension (or gel). At this point, it should be noted that the structural build-up is not the only condition for a system to be thixotropic. In order for a system to be thixotropic, each flow unit composing the network should have a relaxation time[18] of appreciable magnitude so that the time required for the structural reformation is measurable.

* The curves obtained by decreasing the shear rate from 113 $sec.^{-1}$ to 2.3 $sec.^{-1}$

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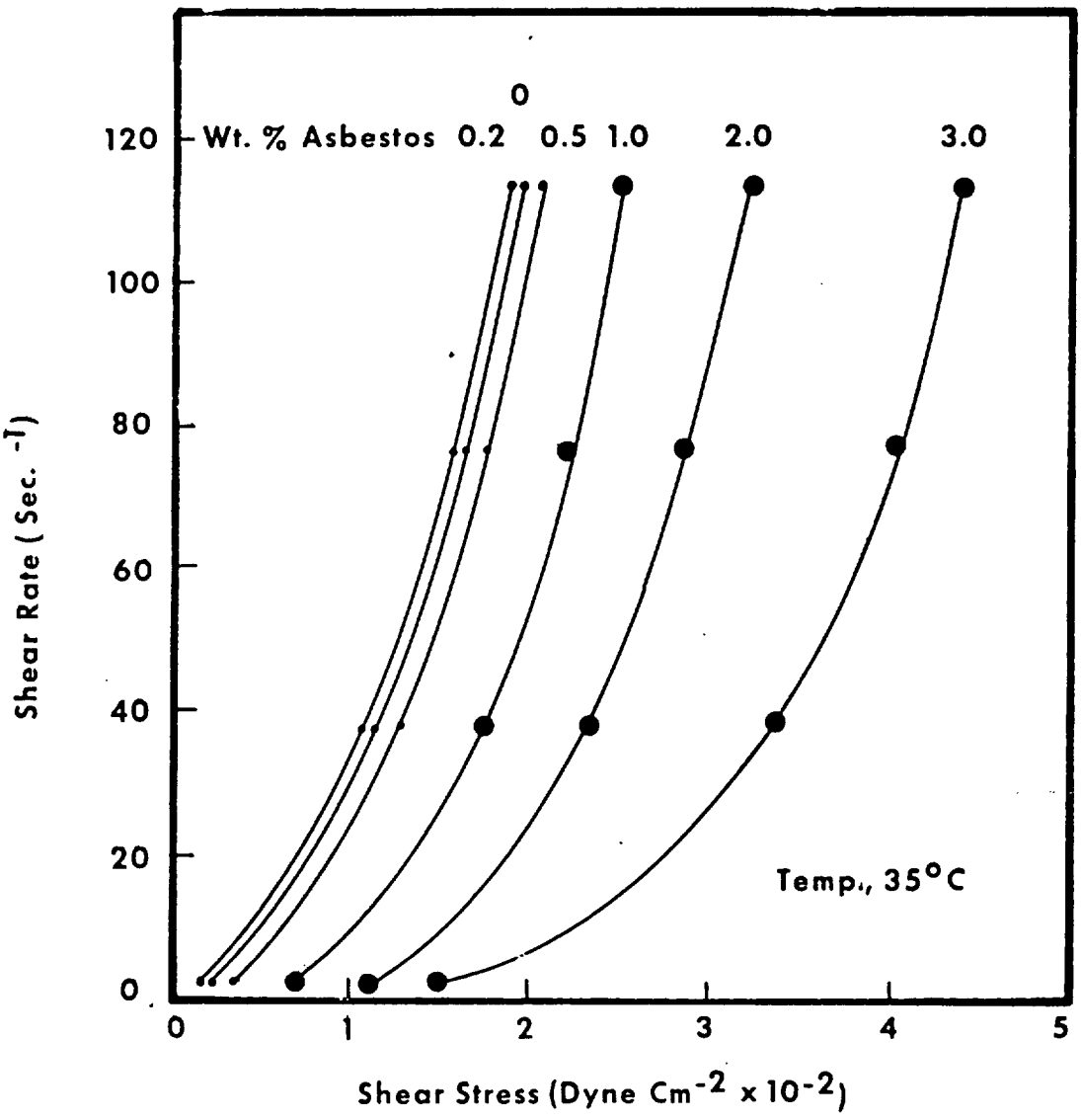


FIG. 7 TYPICAL FLOW CURVES OF ASBESTOS SUSPENSION IN 0.5 WT. % CMC (P-75-HX) SOLUTION FOR SIX DIFFERENT DISPERSOID CONCENTRATIONS.

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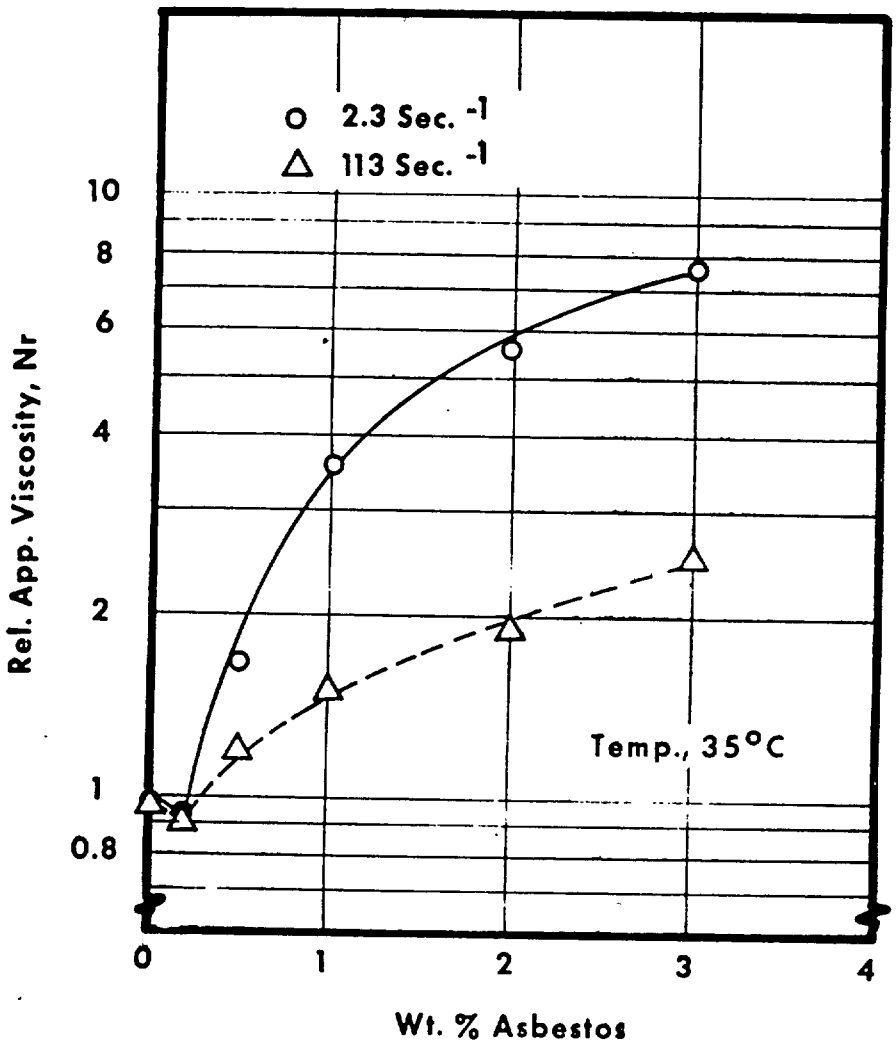


FIG. 8 RELATIVE APPARENT VISCOSITY OF ASBESTOS SUSPENSION IN 0.5 WT. % CMC (P-75-HX) SOLUTION AS A FUNCTION OF DISPERSOID CONCENTRATION.

The Data represents Relative Viscosity Values at Two Different Shear Rates.

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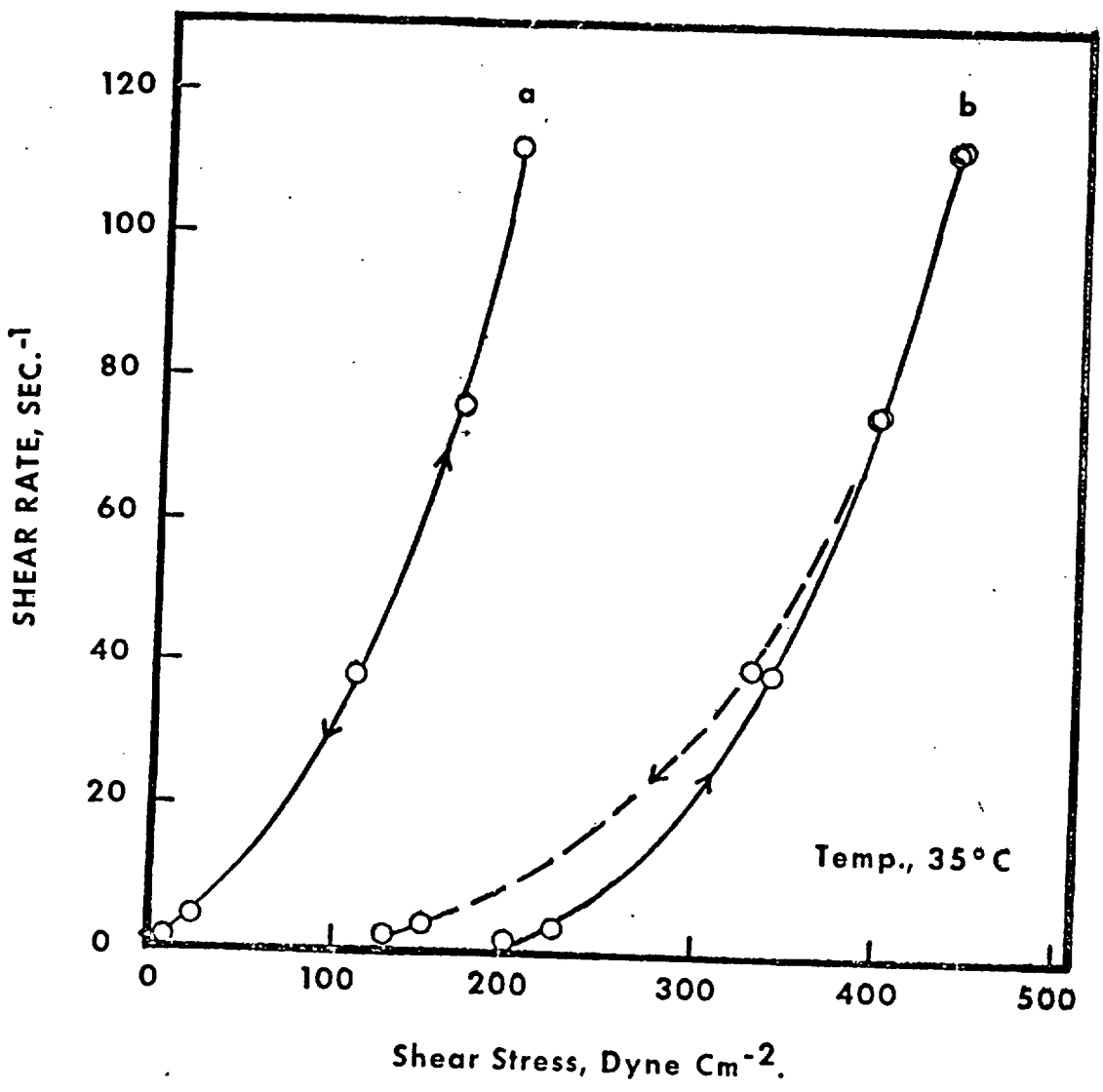


FIG. 9 THIXOTROPIC HYSTERESIS OF ASBESTOS SUSPENSION IN A CMC SOLUTION. a; FLOW CURVE OF 0.5 WT. % AQUEOUS SOLUTION OF P–75–HX. b; FLOW CURVE OF 3% (BY WEIGHT OF TOTAL SUSP.) ASBESTOS SUSPENSION IN 0.5 WT. % P–75–HX. (One cycle ≃ 3 min.)

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II. Non-ionic Polymers

Figures 10 and 11 show the effect of three different non-ionic polymers on the apparent viscosity of 0.5% (by weight total suspension) chrysotile suspensions (pH ≈9). It is seen that Gantrez-M (polyvinylmethyl ether) disperses the chrysotile suspension to some extent. The PVP/VA also appears to disperse chrysotile in 1.5~2.5 pha of polymer addition as supported by visual observation. On addition of PVP/VA (in the vicinity of 2 pha) the surface polarity of chrysotile suspension was reversed from positive to negative. It is not understood whether the charge reversal was rendered by the polymer itself or it occurred due to the presence of some anionic impurities, e.g., hydrolized vinyl acetate which may come with the polymer.

In order to determine if there is any significant amount of ionic impurities, conductivities of aqueous solutions of varying concentrations of PVP/VA were measured. Specific conductivity values varied from 2.5×10^{-6} to 5.0×10^{-6} ohm^{-1} cm^{-1} (at 24°C.) as the polymer concentration increased from 0.1 to 0.4 wt. % (equivalent to 1 to 4 pha in Figure 11). The indication is that the amount of ionic species present in the PVP/VA samples was very small since the specific conductivity of water used for preparing PVP/VA solutions was 2×10^{-6} ohm^{-1} cm^{-1} at 24°C.

In contrast with two above-mentioned non-ionic polymers, no dispersion was observed with PVP. The viscosity of the chrysotile suspension did not vary significantly with increasing the polymer addition (Figure 10). The pH of the suspension essentially did not change as the polymer concentration increased from 0.5 to 8 pha. It is apparent that affinity of PVP for chrysotile is weak compared to the other two non-ionic polymers.

The data in Figure 12 show the effect of sulfate ions on the stability of a dispersed chrysotile suspension to which a varying amount of two different non-ionic polymers was added. The pH of the suspension remained almost unchanged as the polymer concentration increased from 0.5 to 8 pha. Polyox appears to display a protective action, depressing the sensitivity of the chrysotile suspension toward flocculation. It is seen that the pure chrysotile suspension is more flocculated (has the higher viscosity), in the presence of sulfate ions, than the same suspension with Polyox added.

Although some protective action in a chrysotile suspension was observed with Gantrez-M in the flocculation test (see Table II), the viscosity data did not confirm the effect (protecting action). It is seen in Figure 12 that the viscosity value of 1% chrysotile suspension does not change appreciably with Gantrez-M addition, i.e., the suspensions, whether or not they are treated with the polymer, are flocculated to almost the same extent.

Dispersing action (Figures 10 and 11) or protecting action (Table II and Figure 12) displayed by some of the non-ionic polymers for chrysotile asbestos clearly demonstrate that the polymer molecules interact with chrysotile, changing the interfacial property of the latter.

It may first appear that the non-ionic polymer molecules having highly polar groups such as $\rangle C = O$ and -O- (both are strong electron donors) will interact strongly with cationic sites at the surface of chrysotile. However, our results indicate that interaction between such polar molecules and chrysotile

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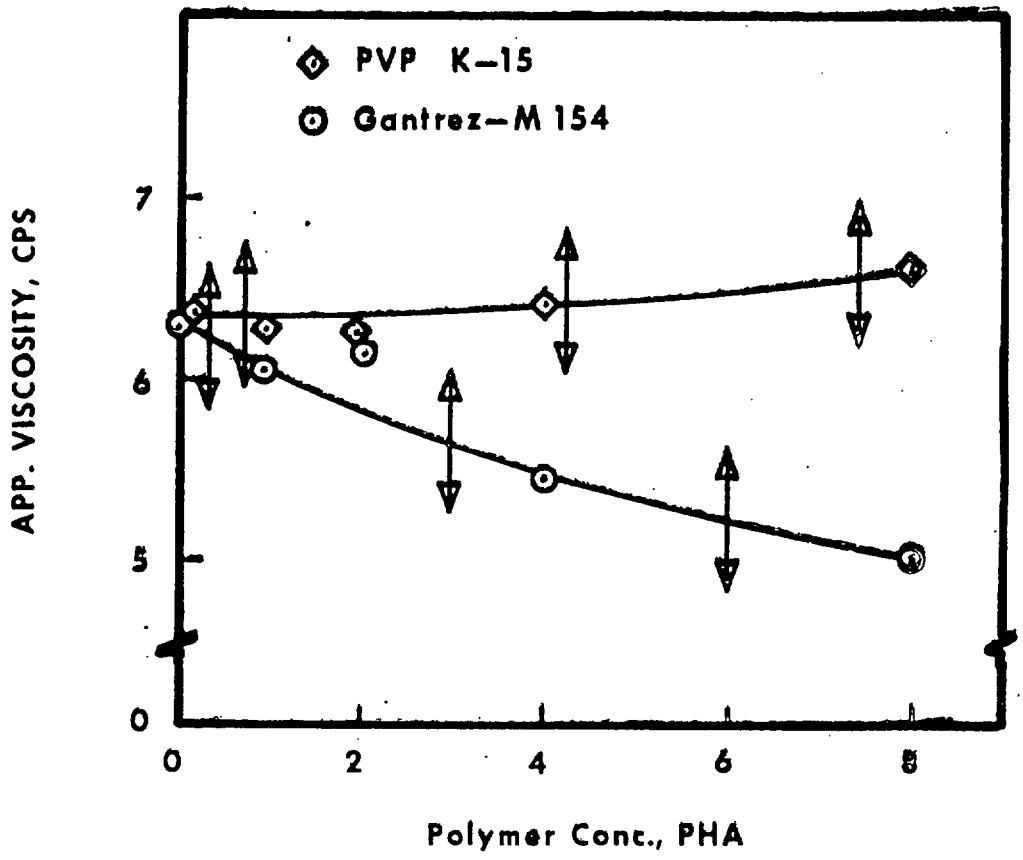


FIG.10 APPARENT VISCOSITY (SHEAR RATE = 511 SEC.$^{-1}$) OF 0.5 WT. % ASBESTOS SUSPENSION AS A FUNCTION OF CONCENTRATION OF TWO DIFFERENT NON-IONIC POLYMERS. PH of the original suspension $\simeq$ 9. Temp., 35°C

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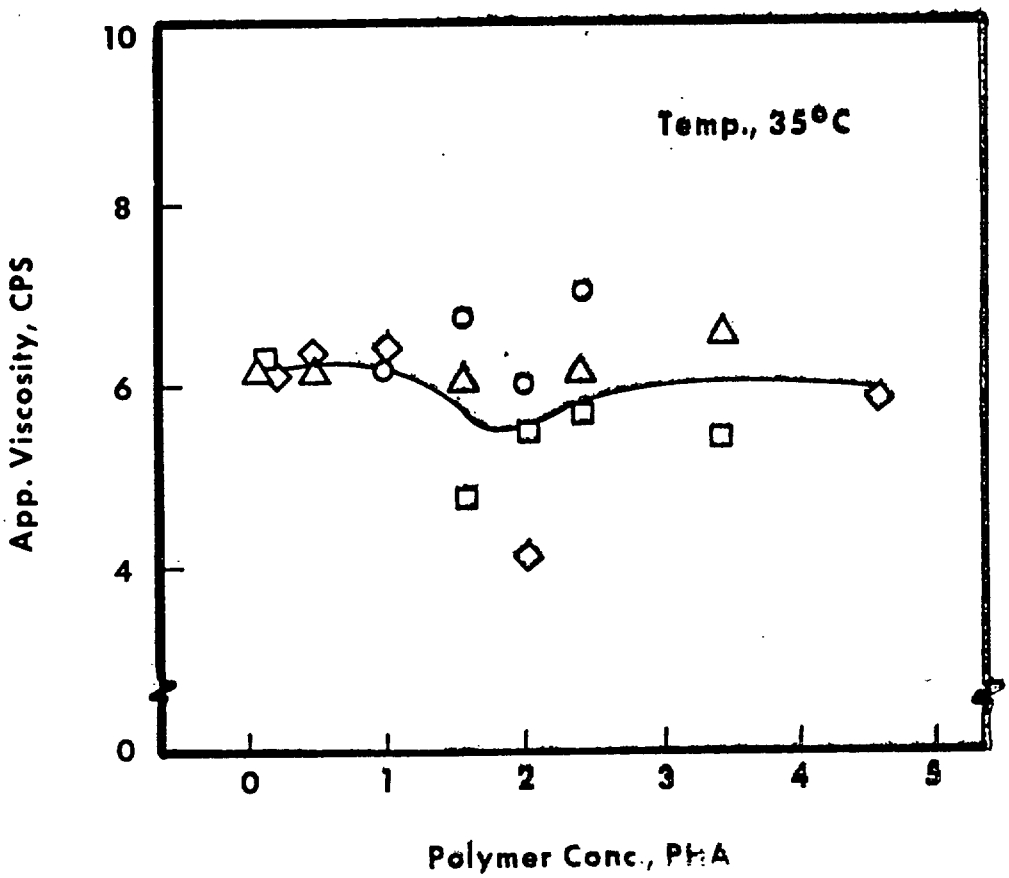


FIG. 11 APPARENT VISCOSITY (SHEAR RATE = 511 SEC.$^{-1}$) OF 0.5 WT. % ASBESTOS SUSPENSION AS A FUNCTION OF PVP/VA CONCENTRATION. PH of the Original Suspension ≒ 9

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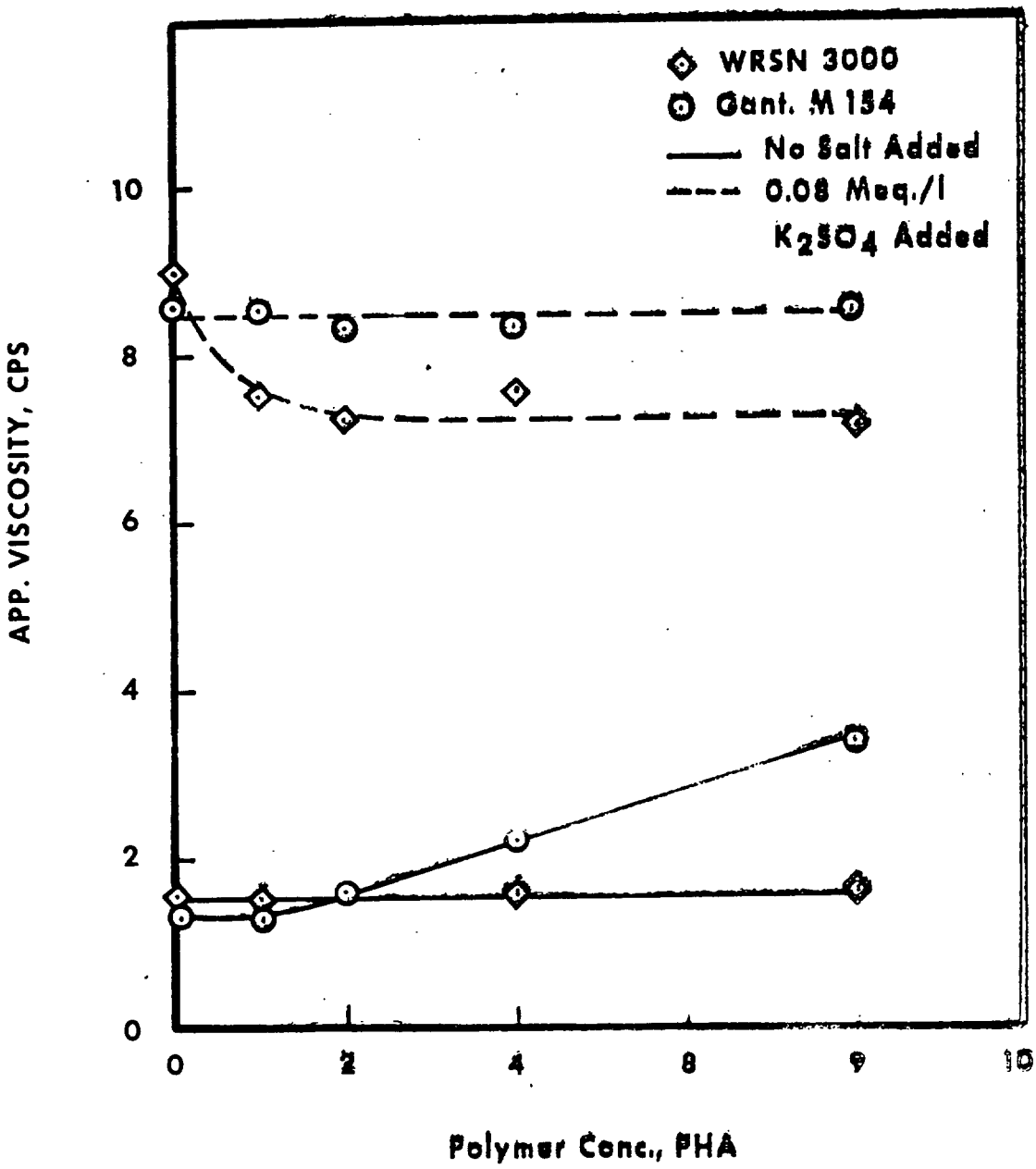


FIG. 12 EFFECT OF TWO NON-IONIC POLYMERS ON THE STABILITY OF 1 WT. % ASBESTOS SUSPENSION (PH = 4.5) IN THE PRESENCE OF SULFATE IONS. SHEAR RATE = 511 SEC.$^{-1}$ TEMP., 35°C.

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asbestos is rather weak. It is seen, from the data presented previously, that dispersing or protecting capacity of non-ionic polymers is much less pronounced compared to that observed with the anionic polymers.

A weak affinity of the non-ionic polymer molecules for chrysotile is understandable when it is realized that those groups ($\rangle$ C = O or -O-) are strongly solvated by water molecules, so that the reactivity of these groups with the polar surface sites of chrysotile becomes weak in the presence of water. Even if it were assumed that chrysotile surfaces have an appreciable number of un-ionized hydroxyl groups, adsorption of polymer molecules onto chrysotile surfaces via hydrogen-bonding does not seem likely because hydrogen-bond adsorption does not generally occur in aqueous solutions when one of the bonding groups has too strong an affinity for water.[19]

However, it also seems possible that adsorption between non-ionic polymers and chrysotile could be promoted by non-polar van der Waals forces. Although there are a number of instances where interaction (e.g., flocculation) takes place between lyophobic colloids and non-ionic polymers,[3] the mechanism for such an interaction is not clearly understood. Again, understanding the true mechanism of interaction between the polymer molecules and chrysotile must be developed from detailed adsorption studies.

Figure 13 shows the typical flow curves of chrysotile suspensions in 1.5 wt. % Polyox solution for four different dispersoid concentrations. The data were again equilibrium curves which were obtained after a prolonged shearing. The curves in general appear to have characteristics of pseudoplastic flow. However, the system did not show any appreciable amount of thixotropy.

Comparing Figures 7 and 13, one sees that consistencies of chrysotile/Polyox suspensions are generally heavier than those of chrysotile/CMC systems.

It was noted (Figure 9) that the chrysotile suspension in CMC solution was thixotropic, and the phenomenon has been attributed to the increased number of flow units forming the spatial structure, which results from adsorption of CMC molecules onto chrysotile particles. However, considering the fact that Polyox molecules have much weaker affinity for chrysotile than do CMC molecules, it is reasonable that formation of thixotropic structure by chrysotile should be more probable in the chrysotile/C M C suspension than in the chrysotile/Polyox systems.

III. Cationic Polyelectrolyte

The effect of a typical cationic polyelectrolyte on the apparent viscosity of 0.5% (by weight of total suspension) chrysotile suspension is shown in Figure 14. Polymer, X-150 is poly (1,2-dimethly-5-vinyl pyrridinium), methyl sulfate.

It is seen that the viscosity rises markedly as the amount of polymer added increases.* However, in a suspension of lower dispersoid concentration, e.g., 0.5 wt. %, the apparent viscosity remained almost unchanged while the amount of polymer added was increased from 0.15 to 1.2 pha (data not presented).

* 4 pha in Figure 14 is equivalent to 0.01% by weight of water in the suspension.

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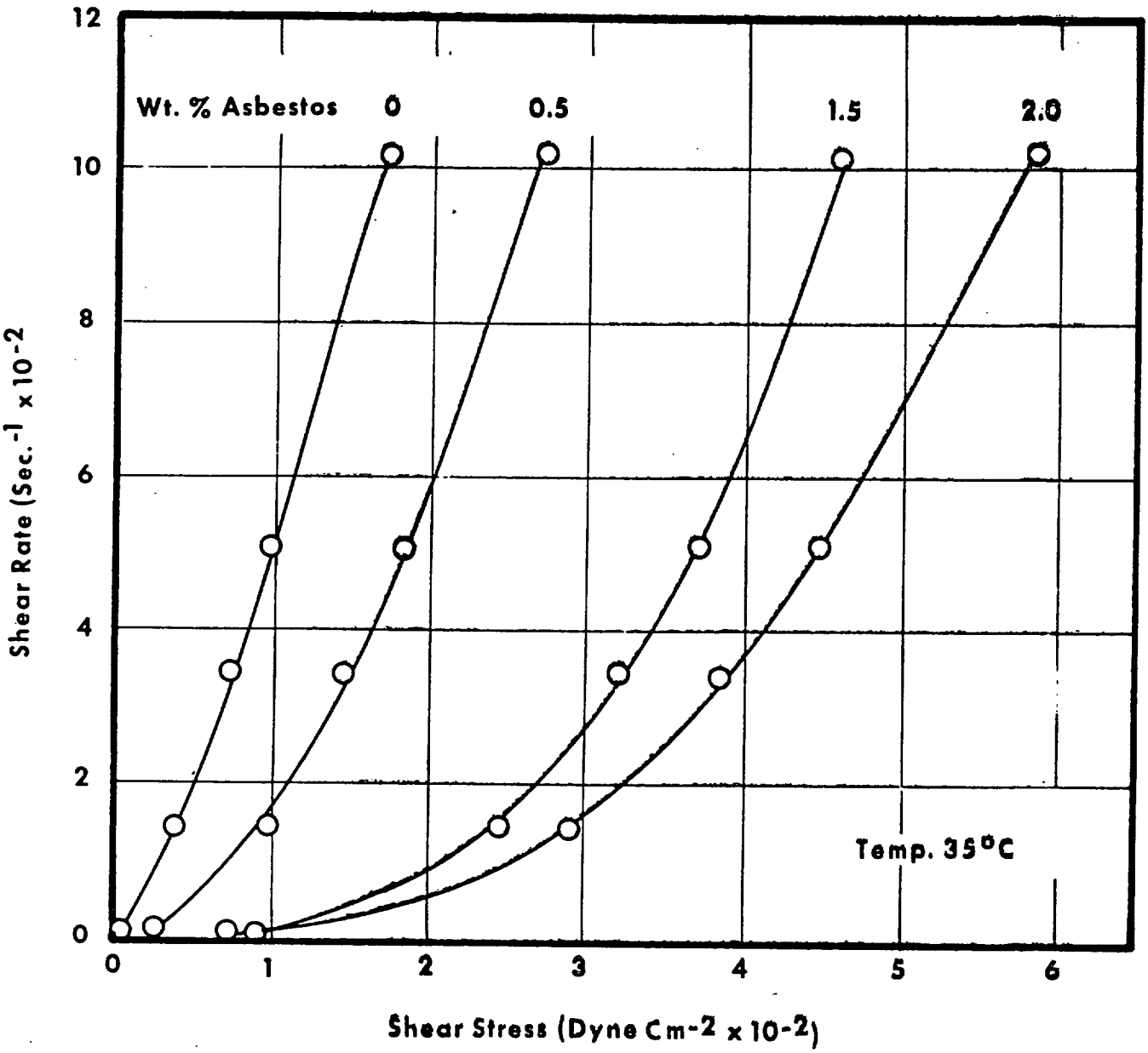


FIG. 13 TYPICAL FLOW CURVES OF ASBESTOS SUSPENSION IN 1.5 WT. % POLYOX (WSRN-3000) SOLUTION FOR FOUR DIFFERENT DISPERSOID CONCENTRATIONS.

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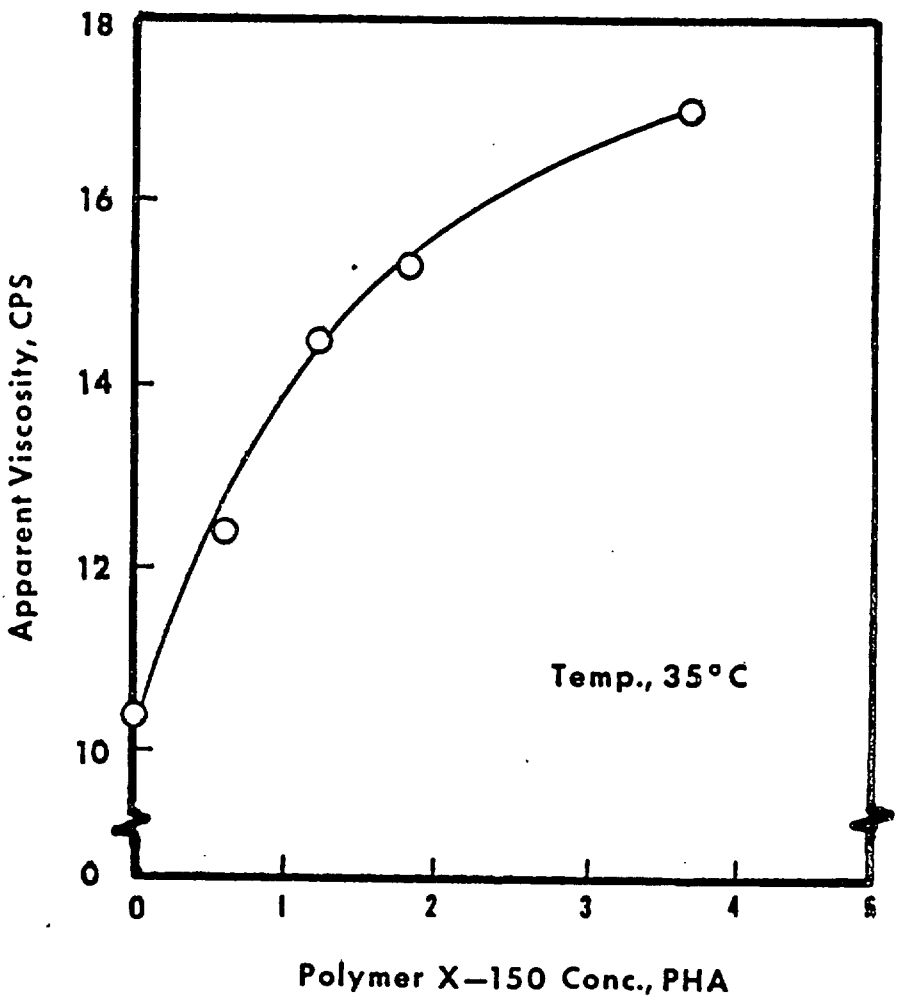


FIG.14 EFFECT OF CATIONIC POLYMER ON APPARENT VISCOSITY (SHEAR RATE = 511 SEC.$^{-1}$) OF 1.0 WT. % ASBESTOS SUSPENSION. PH of the Original Suspension $\simeq$ 9

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Because of the fact that the surface of chrysotile is essentially electropositive and the polyelectrolyte molecules carry the similar charges, it would be expected that introduction of the latter into a chrysotile suspension will merely result in an increase in hydrodynamic volume, which should cause some increase in viscosity. Then, the large increase in viscosity with polymer addition may be attributed to the unique flow characteristics of the cationic polyelectrolyte itself. In his study on viscosities of dilute aqueous solutions of quaternized poly-4-vinyl pyridine, Eisenberg[20] found that the plot of viscosity number, $\frac{\eta_{sp}}{\varphi}$ against the concentration, φ, showed strong upward curvature, suggesting that unusually strong interaction occurs among the molecules of the cationic polyelectrolyte. Such a strong interaction is generally ascribed to a stretching of the polyelectrolyte molecules due to electrostatic repulsive forces between similar charges on the chain.[21] Thus, the interaction between chrysotile particles and the stretched polyelectrolyte molecules, together with the strong repulsion among the molecules themselves, would cause the marked increase in viscosity.

However, there may be other factors which could contribute to the pronounced increase in viscosity, such as flocculation of chrysotile promoted by adsorption of cationic polyelectrolyte molecules. Inasmuch as there is an indication that some negative sites are present on the chrysotile surfaces,[22] it is probable that some chrysotile (or aggregates) may be held together by a polyelectrolyte molecule part of which is adsorbed on the negative sites of chrysotile surfaces (bridging mechanism).

CONCLUSIONS

The information obtained should make it possible to determine the basic variables which influence the stability of chrysotile asbestos in aqueous solutions of water-soluble polymers in general. It was found that in order to obtain a stable thixotropic system from chrysotile asbestos suspended in a polymer solution, the polymer must disperse chrysotile, increasing the number of chrysotile particles (either individual fibers or fiber bundles), which favors formation of a spatial network. Such a dispersion occurs if the polymer molecules have strong affinity for chrysotile; thus, they are adsorbed onto chrysotile surfaces. On the other hand, if the polymer molecules have either a weak affinity or they do not have affinity at all for the chrysotile surface, the system tends to show time-independent pseudoplastic flow characteristics.

The present investigation also provides valuable information for modifying the chrysotile surface so that it exhibits desirable properties in various other liquid media (polar and non-polar). It has been found that chrysotile products having a wide range of surface polarity or chemical reactivity can be made by treating chrysotile with water-soluble polymers.

Some of the potential applications of chrysotile asbestos in connection with water-soluble polymers are as follows.

(1) Asbestos as a Dispersoid in Aqueous Solution of Water-Soluble Polymers

Chrysotile is an effective suspensoid in many water-soluble polymers. A stable thixotropic system can be obtained from a solution of anionic polyelectrolytes, in which chrysotile is dispersed. A solution of non-ionic polymers (possibly cationic polymers as well) thickened with chrysotile asbestos appears to exhibit the time-independent pseudoplastic flow characteristics.

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(2) Water-Soluble Polymers as Protective Colloids or as Flow Modifying Additives in Chrysotile Aqueous Suspension

Since the anionic polyelectrolytes are capable of dispersing or flocculating a chrysotile suspension, they seem to be versatile flow modifying additives for an aqueous chrysotile suspension. Furthermore, most anionic polyelectrolytes function as protectice colloids for chrysotile suspensions. For instance, an asbestos suspension treated with hydrolyzed poly (ethylene/maleic anhydride) remained very stable when sulfate ions were added, which normally flocculate chrysotile. A chrysotile suspension which is stable in a wide pH range can be provided by treating chrysotile with a certain anionic polyelectrolyte. Although their capacity is small, some non-ionic polymers such as Polyox (polyethylene oxide) also shows protecting action.

In order that asbestos can be introduced as a drilling fluid dispersoid, it is necessary to develop chrysotile products having good flow and filtration properties (low filtration rate) as well as good stability in the presence of various ionic species. Such a chrysotile product could be provided by treating chrysotile with a proper form of anionic polyelectrolyte. For instance, a pretreatment of chrysotile with CMC (sodium carboxymethyl cellulose) improved significantly the filtration property of an aqueous suspension containing asbestos and bentonite, i.e., the filtration rate as well as the filter cake thickness was considerably reduced when chrysotile was treated with the polymer before it was admixed with the bentonite.

(3) Modified Asbestos Products

A number of asbestos products whose surface properties are very different from the natural products can be provided by adsorption of water-soluble polymers. For instance, chrysotile asbestos having strongly negative surface charge can be prepared by treating chrysotile with a small amount (c., 2 parts per hundred of asbestos) of anionic polyelectrolyte. Inasmuch as the water-soluble polymers (or their derivatives) of a wide range of polarity or chemical reactivity are available, chrysotile products of versatile surface property could be provided.

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APPENDIX

List of Water-Soluble Polymers Used

Trade Name	Chemical Name	Charge Type	Manufacturer	Ref.*
Cellocize, P-75-HX	Sodium carboxymethyl cellulose	anionic	Union Carbide	11**
Cellex (CMC)	"	"	Baroid	-
EMA-11	Poly (ethylene/maleic anhydride)	"	Monsanto	1-261
Carbopol-941	A polyacrylic acid(?)	"	B. F. Goodrich	GC-36
Gantrez-An 169	Poly (methyl vinyl ether/maleic anhydride)	"	General Aniline and Film	7543-017
Gantrez-An 4151	Ammonium salt of half amide of poly (methyl vinyl ether/maleic anhydride)	"	"	1260-66
Gantrez-M-154	Poly (vinyl methyl ether)	non-ionic	"	7543-078
PVP K-15	Polyvinyl pyrrolidone	"	"	7543-113
PVP/VA	Copolymer of PVP and vinyl acetate	"	"	7543-031
Polyox WRSN-3000	Poly (ethylene oxide)	"	Union Carbide	F-40246D
Polymer X-15	Poly (1,2-dimethyl-5-pyrridinium), methyl sulfate	cationic	"	F-41177

* Technical bulletin provided by the manufacturer
** References cited in this report

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