

## Rheological Properties of Carbomer Dispersions

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### ABSTRACT

Rheological properties of carbomer solutions were investigated as a function of carbomer concentration. Carbomer is an emulsion stabilizer. The shear stress was measured as a function of shear rate and shearing time. Both dynamic and static yield stress,  $\tau_0$ , values were also measured. It was found that below 0.45 wt% of carbomer content, carbomer dispersion behave as a Newtonian fluid, whereas at or above 0.45 wt%, the dispersions exhibit a shear-thinning and rheopectic behaviour. The yield stress results are in harmony with the shear stress- rate experiments that below 0.45 wt%, a zero-yield stress value was reported and a non-zero value at or above that point.

### INTRODUCTION

Carbomer is a fine white acrylic powder used in hair gels, and other gels, lotions, and creams. It is suitable for formulating sparkling and clear gels as well as stabilizing emulsions. The typical use level is 0.1-0.5% depending on the type of formulation and final desired viscosity. It is a cost effective thickener and is pH sensitive. Carbomer is a water and alcohol soluble polymer. It is a mildly acidic and forms a loose network when partially neutralized to pH 5-8. Carbomer requires a long time to swell properly for use. It is best to make a pre-gel before beginning the batch

at hand. It can be used in cosmetic products, like lotions, gels, and hair products.

Moreover, the use of bio-adhesive polymers like carbomer for ocular drug delivery was the subject of several studies published during the last decade and several review articles are available<sup>1</sup>. Carbomer, a polyacrylic acid polymer, is available in a wide range of molecular weights and either being linear, branched or cross-linked<sup>2</sup>. The acidic carboxyl groups partially dissociate in water, producing a flexible coil. It is thought, upon the addition of alkaline compounds, solvation, salt formation and electrostatic repulsion between the anionic groups cause gel formation<sup>3</sup>.

Simovic et al.<sup>4</sup> investigated the system made of carbomer and a non-ionic surfactant, with the aim of achieving better understanding of emulsion stabilization mechanisms. A 0.2%w/w aqueous dispersion of polymeric emulsifier, containing various amounts of surfactant (from 0.01 to 1.0% w/w) was used throughout their study. For the hydrophobic, modified carbomer, they reported an increase in viscosity and texture profile parameters with increasing Polysorbate 80 concentration up to 0.3% w/w, followed by a decrease in the gel network strength at higher surfactant concentrations. On the other hand, the unmodified, hydrophilic carbomer (Carbopol 934P®) showed small changes in the network structure with the

increasing concentration of surfactant; hence the interactions between emulsifier and surfactant are of a hydrophobic nature, concentration-dependent, and affect the rheological properties of dispersion.

Rheological properties of carbomer systems were found to affect the time-dependent release or the bioavailability of the active ingredient being used in drug delivery systems. For example, Esposito et al.<sup>5</sup> investigated the influence of liposomal formulations on the in vitro absorption of methyl nicotinate (MN), which was taken as model drug. Their results indicated that MN permeability was directly related to soybean phosphatidylcholine concentration and inversely related to liposome size and to vehicle viscosity.

In this research, the rheological properties of carbomer dispersion, with varied concentration, will be examined. The shear stress vs. shear rate and vs. shearing time, in addition to the yield stress measurement will be exploited. Viscosity and yield stress measurements are useful in telling the effects of processing, formulation changes, and aging phenomena.

## EXPERIMENTAL METHODS

### Materials

Carbomer, type 940, carboxyvinyl polymer; average equivalent weight: ca.76; bulk density: 0.20-0.23 g/cm<sup>3</sup>; pH (0.5% water dispersion): 2.7-3.5 was used. It was purchased from The Personal Formulator Company,

<http://thepersonalformulator.com>

Wyoming; U.S.A. Carbomer must be thoroughly mixed and hydrated. Increasing the pH to 7.0, gives a gel structure. Neutralization can be carried out with inorganic bases (such as NaOH, KOH, NH<sub>4</sub>OH) or with organic amines, such as Tri-Ethanol-Amine (TEA), Amino-Methyl-Propanol (AMP), and Amino-Methyl-Propane-Diol (AMPD). To neutralize 1 g of Carbomer to pH 7, ca. 0.01 equivalent of base are required (e.g. 0.4g of NaOH, 0.9g

of AMP, 1.5g of TEA). It is advisable to add strong bases previously diluted into water at a concentration not higher than 10%.

### Sample Preparation

Carbomer requires a long time to swell properly for use. It is best to make a pre-gel before beginning the batch at hand. Consequently, carbomer, type 940, was weighted and added to a known volume of distilled water at room temperature. The sample was left for 24 hours at room temperature. The next day, the sample was stirred to assure homogeneity and dispersity of carbomer in the aqueous solution. The following concentrations of carbomer were used: 0.15, 0.30, 0.45, 0.6, and 0.90 wt %.

### Accuracy of Viscosity Measurements

The Brookfield LDV-III Ultra Programmable Rheometer was used to measure fluid parameters of shear stress and viscosity at given shear rates. The principle of operation of the LDV-III Ultra is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. The viscosity measurement range of the LDV-III Ultra (in centipoises or cP) is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full scale torque of the calibrated spring. The LDV-III Ultra can also measure yield stress (in Pascal or Pa).

Two spindles were used for measurement of viscosity of carbomer dispersions: LV#1, with an entry code of 61, and LV#2, with an entry code of 62. For LV#1 spindle, the spindle range coefficient is 6,000 while for LV#2 spindle it is 30,000. Such numbers are used to evaluate the full scale viscosity range (FSVR) for a rheometer/spindle/speed combination. The accuracy of viscosity measurement for the LDV-III Ultra rheometer is  $\pm 1.0\%$  of its full

scale range for a specific spindle running at a specific speed.

Instrument Accuracy for  $\eta$  Measurement,

$$IA_{\dot{\gamma}} = \frac{0.01 * 6,000}{RPM} cP (mPa.s) \quad (1)$$

$IA$  is subscripted by  $\dot{\gamma}$  to indicate that the instrument accuracy is a function of the spindle speed, RPM (i.e.,  $\dot{\gamma}$ ). The higher the value of  $IA_{\dot{\gamma}}$  is, the lesser the accuracy of the instrument at the given spindle speed will be. For example, if LV#1 spindle is run at 10 rpm, then  $FSVR=6,000/10=600$  cP (mPa.s) and the accuracy of measurement will be  $\pm 0.01 * 600 = \pm 6$  cP (mPa.s). For LV#2, 6,000 is replaced by 30,000.

#### Calibration of Brookfield LDV-III Ultra Programmable Rheometer for Viscosity, Shear Stress, and Shear Rate Measurements

According to the operating instructions given in Brookfield Manual number M/98-211-B0104, Appendix D1, pages 94-97, Table 1 shows the results for finding the spindle multiplier constant (SMC) which is used to calculate cP values and the shear rate constant (SRC) which is used to calculate shear rate and shear stress. SMC and SRC values were found for a particular cylindrical container that has an inside radius of 2.2175 cm (22.175 mm) while using the spindle LV1 with entry code 61 which has a radius of 0.942 cm (9.42 mm). The LDV-III Ultra torque constant (TK) is given as 0.09373 (Table D2 of Brookfield manual). The 5,000-standard solution, provided by Brookfield Engineering Laboratories, has  $n$  value of 4940 cP. The same procedure of calibration was

repeated for the other three spindles provided by the manufacturer.

#### Apparent Viscosity Measurement

After the calibration is done, each sample is subjected to a variable shear rate (i.e., different RPM) both in the forward (increasing) and backward (decreasing) shear rate starting at one point, followed by a stepwise increment in RPM value, then followed by a stepwise decrement in RPM value till the starting point is reached. At each step 30 seconds was allowed for the sample to reach its steady value at the given RPM. As far as the shearing time effect is concerned, the sample was subjected to a constant shear rate for 4 minutes to see if there is a time-dependent flow behavior. The measurement was taken for each 30 second elapsing period. If the torque reading is beyond the upper limit (i.e., above 100% torque reading), then the spindle is replaced by a smaller spindle size to assure that the torque reading lies within the range (i.e., between 10 and 100% torque reading, in general).

#### Yield Stress Measurement

For the yield stress measurement, LDV-III is equipped to measure yield stress vs. yield strain. The yield point is the point at which a material begins to flow. The associated properties are the yield stress and yield strain. The yield stress is the critical shear stress, applied to the sample at which the material begin to flow as a liquid. The yield strain is the deformation in the material, resulting from the applied stress prior to the start of flow. The operating principle is to drive a vane spindle through the calibrated spiral spring connected to a motor drive shaft. The vane spindle is immersed in the test material. The vane spindles are suitable for most fluids and are ideal for paste-like materials, gels, fluids

with suspended solids, and a variety of so-called “soft solid” materials (puddings, sauces). The primary benefit of the vane spindle is that it imparts minimal disruption to the sample during spindle immersion. The resistance of the material to movement is measured by observing increasing torque values as the LDV-III Ultra motor rotates. The amount of shaft rotation is measured by the deflection of the calibrated spiral spring inside the instrument. Spring deflection is measured with a rotary transducer. There is

usually some deformation of the test material due to the increasing force imparted by the vane spindle. The maximum torque value is the yield point. An algorithm in the firmware converts the maximum torque value into a yield stress value, expressed in Pascal (Pa).

Table1: Calculation of SMC and SRC for a particular set of spindle/container.

Spindle LV#1 with entry code of 61 was used in a plastic cylinder with a radius of 22.175 mm						
RPM	Y(Torque % reading)	$R1 = \frac{100n}{Y}$	$SMC = \frac{R1 * RPM}{0.09373 * 10,000}$	$R_c^2$	$R_b^2$	$SRC = \frac{2(2\pi/60)R_c^2R_b^2}{R_b^2[R_c^2 - R_b^2]}$
0.2	17.2	28720.93	6.128439	4.9173	0.8874	0.25556
0.5	41.3	11961.26	6.380699			0.25556
1	81.1	6091.245	6.498715			0.25556
Average			6.335951			0.25556

$$\dot{\gamma} = SRC \times RPM \quad \eta = \frac{Y}{100} \times FSVR = \frac{Y}{100} \times 0.09373 * \frac{10,000}{RPM} * SMC$$

Note:  $\eta = n$  for a standard solution.

#### Accuracy of Yield Stress Measurement

The accuracy of measurement for yield stress values is defined as follows:

Instrument Accuracy for  $\tau$  measure.,  
 $IA_{\tau} = 0.01 \times FSYSR$  (2)

where FSYSR stands for Full Scale Yield Stress Range, expressed in Pascal (Pa).

$$FSYSR = TK \times YMC \times 10$$

$$= 0.09397 \times YMC \times 10$$
 (3)

where YMC is the yield multiplier constant which is given in Table 2. Thus:

Table 2: Spindle codes used in yield stress tests.

Spindle	YMC	Vane Length (cm)	Vane Diameter (cm)
V-71	0.5	6.878	3.439
V-72	2.0	4.333	2.167
V-73	10	2.535	1.267

Note: If the secondary immersion mark is selected, the YMC value is multiplied.

For example, if spindle V-71 is used while the spindle is completely immersed up to the primary mark, then  $IA_{\tau} = \pm 0.01 * 0.09397 * 0.5 * 10 = \pm 4.7 \times 10^{-3}$  Pa ( $\pm 4.7$  mPa).

$$IA_{\dot{\gamma}} = 9.397 \times 10^{-3} \times YMC \quad (4)$$

## RESULTS AND DISCUSSION

### The Apparent Viscosity, $\eta$ Results

For 0.15 wt% carbomer dispersion, the apparent viscosity value was found to be  $7.8 \pm 0.2$  cP where 0.2 represents the standard deviation for  $\eta$  under the variable shear rate,  $\dot{\gamma}$ , experiment. The smallest value of  $IA_{\dot{\gamma}}$  is 0.32 cP. This simply implies that within the instrument accuracy ( $IA_{\dot{\gamma}} = 0.32$  cP or above) and with such a low concentration of carbomer, the dispersion behaves well as a Newtonian fluid where the viscosity remains essentially constant with  $\dot{\gamma}$ . Figure 1 shows the plot of  $\eta$  vs.  $\dot{\gamma}$  at such a level of carbomer content. One can see how the distribution of data falls within the margin of the instrumental accuracy value.

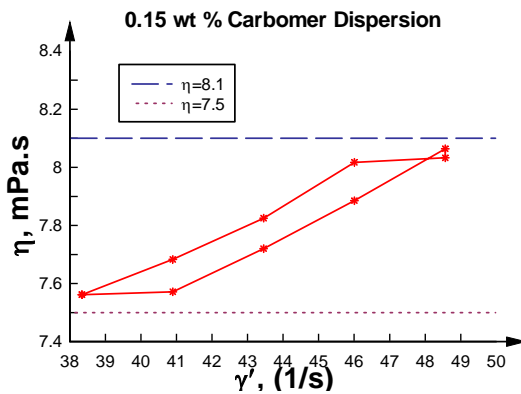


Figure 1: The apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , ( $s^{-1}$ ) for 0.15 wt% carbomer dispersion, measured at  $T=22^{\circ}C$  with LV#1 spindle in a plastic cylinder with a radius of 22.175 mm. The  $IA_{\dot{\gamma}}$  margin is bounded between the two horizontal lines.

For 0.30 wt% carbomer dispersion, the apparent viscosity has an average value of  $15.06 \pm 0.51$  cP. For the variable shear rate

experiment,  $IA_{\dot{\gamma}}$  varied between 0.67 and 1.2 cP, with a mean  $IA_{\dot{\gamma}}$  value of 0.95 cP. In this case, carbomer dispersion up to this concentration and within the instrumental accuracy may be treated as a Newtonian fluid. Figure 2 shows the plot of  $\eta$  vs.  $\dot{\gamma}$  at such a level of carbomer content. Again, one can see how the distribution of data falls within the margin of the instrumental accuracy value.

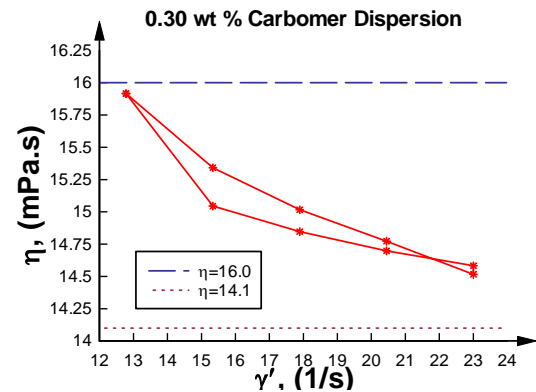


Figure 2: The apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , ( $s^{-1}$ ) for 0.30 wt% carbomer dispersion. Other settings are the same as those of Fig. 1. The  $IA_{\dot{\gamma}}$  margin is bounded between the two horizontal lines.

For 0.45 wt% carbomer dispersion, the apparent viscosity has an average value of  $107.7 \pm 28.5$  cP. For the variable shear rate experiment,  $IA_{\dot{\gamma}}$  varied between 2.8 and 12.0 cP, with a mean  $IA_{\dot{\gamma}}$  value of 7.4 cP. In this case, the variation in viscosity with  $\dot{\gamma}$  is significant at this stage and it is about  $28.5/7.4=3.8$  times broader than the instrumental accuracy margin, then one can argue that the viscosity is no longer a constant with  $\dot{\gamma}$  and the carbomer dispersion started to behave as a non-Newtonian fluid. Thus, there is a necessity to analyze the data in the light of this finding.

Figure 3 shows the apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , for 0.45 wt% carbomer dispersion. It can be seen that  $\eta$  decreases with increasing  $\dot{\gamma}$ ; this type of behaviour is referred to as a pseudo-plastic or shear-thinning behaviour, which can be seen in paints, emulsions, and dispersions of many types. Such a behaviour may be attributed to the effect of shearing on the structure of carbomer molecules in a way they will be destroyed and the molecule formation will be oriented more parallel to the spindle surface. So, the hindering of the spindle rotation will decrease. The faster the rotation will become, the more the structure damage is done and the less the structure of molecules slide in together, the lower the viscosity will be.

As far as the time-dependent behaviour is concerned, the apparent viscosity remained almost constant with shearing time within the instrumental accuracy range; an indication that at such a concentration or less there is no significant rheopectic (an increase of  $\eta$  with shearing time) or thixotropic (a decrease of  $\eta$  with shearing time) behaviour for carbomer dispersion. Moreover, Figure 3 shows some kind of a hysteresis loop; i.e., the forward and backward directions do not exactly coincide, nevertheless, this can be assumed insignificant if looked upon taking into account the instrumental accuracy. Here, the largest difference in  $\eta$  value between the forward and backward directions  $\Delta\eta = 5.94$  cP which is definitely less than the mean value of  $IA_{\dot{\gamma}}$  under the given shear cycle.

For 0.60 wt% carbomer dispersion,  $\eta$  has an average value of  $1197.1 \pm 1006.26$  cP. For the variable shear rate experiment,  $IA_{\dot{\gamma}}$  varied between 11.65 and 171.40 cP with a mean value of 91.5 cP, which implies that the variation in  $\eta$  with  $\dot{\gamma}$  is very significant. Figure 4 shows  $\eta$  as a

function of  $\dot{\gamma}$  for 0.60 wt% carbomer dispersion.  $\eta$  decreases with increasing  $\dot{\gamma}$ ; an indication of a pseudo-plastic or shear-thinning behaviour.

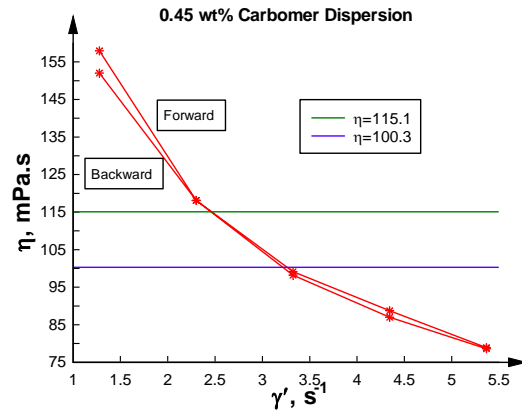


Figure 3: The apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , ( $s^{-1}$ ) for 0.45 wt% carbomer dispersion. Other settings are the same as those of Fig. 1 The  $IA_{\dot{\gamma}}$  margin is bounded between the two horizontal lines.

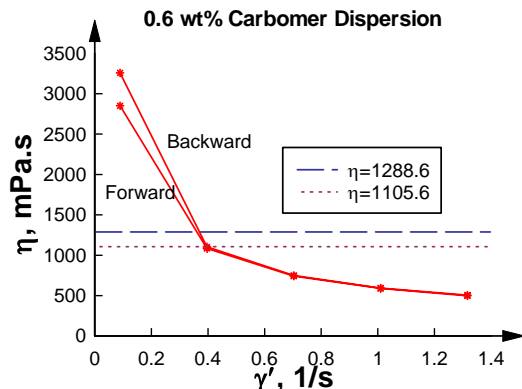


Figure 4: The apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , ( $s^{-1}$ ) for 0.60 wt% carbomer dispersion. Other settings are the same as those of Fig. 1.

The hysteresis loop is now more obvious than that in Figure 3. and the viscosity difference between the forward and backward direction values is larger

than the corresponding value of  $IA_{\dot{\gamma}}$  evaluated at the same RPM (or  $\dot{\gamma}$ ). For example,  $\Delta\eta$  (between forward and backward direction values) evaluated at RPM=0.35 ( $\dot{\gamma}=0.09 \text{ sec}^{-1}$ ) is equal to 3257.77-2850.55=407.22 cP which is about 2.4 larger than the corresponding  $IA_{\dot{\gamma}}$  value ( $IA_{\dot{\gamma}}$  equal to 171.4 cP at RPM=0.35).

This leads us to the conclusion the pseudo-plastic effect is prominent at such level of carbomer concentration. At the same time, the rheopectic behaviour which can be expressed in terms of the viscosity difference between shear rate forward and backward direction values is obvious at low RPM (or low  $\dot{\gamma}$ ) values. Data regarding the effect of shearing time on  $\eta$  are not presented here, simply to make the story short, nevertheless,  $\eta$  slightly increased with shearing time; an indication of rheopectic behaviour. Thixotropic behaviour is usually attributed to the breakdown/alignment of polymer chains or segments, whereas rheopectic behaviour may be attributed to the build-up/entanglement of polymer chains or segments<sup>6</sup>. Shear-thinning and rheopectic (or, non-thixotropic) behaviour of the pharmaceutical properties of estradiol gels made with different carbomers were also found by Amsellem et al.<sup>7</sup>.

For 0.90 wt% carbomer dispersion,  $\eta$  has an average value of  $\eta=1881.14\pm 223.61$  cP. For the variable shear rate experiment,  $IA_{\dot{\gamma}}$  varied between 25 and 37.5 cP with a mean value of 31.2 cP. That implies that the variation in  $\eta$  with  $\dot{\gamma}$  is very significant. Figure 5 shows  $\eta$  as a function of  $\dot{\gamma}$  for 0.90 wt% carbomer dispersion.  $\eta$  decreases with increasing  $\dot{\gamma}$ ; an indication of a pseudo-plastic or shear-thinning behaviour. The hysteresis loop is wide and extends over the entire range of  $\dot{\gamma}$ . The viscosity

difference between the forward and backward direction values is larger than the corresponding value of  $IA_{\dot{\gamma}}$  evaluated at the same RPM (or  $\dot{\gamma}$ ). For example,  $\Delta\eta$  evaluated at RPM=8 ( $\dot{\gamma}=2.04 \text{ sec}^{-1}$ ) is equal to 2234.2-2178.1=56.1 cP which is about 1.5 larger than the corresponding  $IA_{\dot{\gamma}}$  value ( $IA_{\dot{\gamma}}$  equal to 37.5 cP at RPM=8). For the constant shear rate experiment,  $\Delta\eta=101.8$  cP which is obviously larger than  $IA_{\dot{\gamma}}=30$  cP, evaluated at RPM=10 while using spindle LV#2 at this time, unlike previous cases where LV#1 was used.

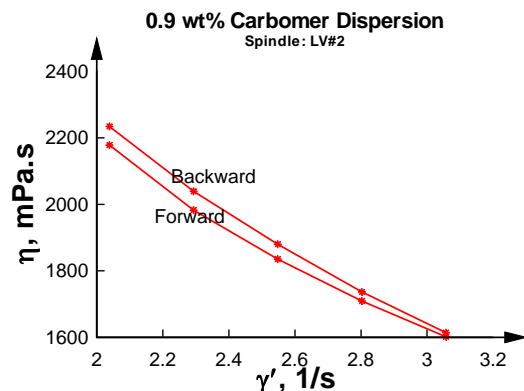


Figure 5: The apparent viscosity,  $\eta$ , as a function of shearing rate,  $\dot{\gamma}$ , ( $\text{s}^{-1}$ ) for 0.90 wt% carbomer dispersion, measured at  $T=22^\circ\text{C}$  with LV#2 spindle in a plastic cylinder with a radius of 22.175 mm.

#### The Yield Stress, $\tau_o$ , Results

For carbomer concentrations of 0.15 and 0.30 wt %, no yield stress could be found for such dispersions, which is in line with what was found earlier in the apparent viscosity measurements. Figure 6 shows a typical plot for the shear stress versus strain for 0.45 wt% carbomer dispersion, where the sample was pre-sheared for 30 s at RPM=10 and the test was carried out at RPM=5, using spindle LV#71, half immersed. The yield stress,  $\tau_o$ , with pre-shearing is  $0.17\pm 0.009$  Pa.

The same experiment was repeated but without a pre-shearing step and the yield stress,  $\tau_o$ , without pre-shearing is  $0.18\pm 0.009$  Pa. Pre-shearing is the shearing of sample before measuring its yields properties. This process beaks down the sample's structure. It is particularly useful if the investigator wants to eliminate previous shear history (e.g., bumping, transferring) of the sample before testing and observe the structural rebuilding of the sample. The yield stress measured in a pre-shear sample is the "dynamic yield", while the yield stress measured for an originally un-disturbed material is the "static head".

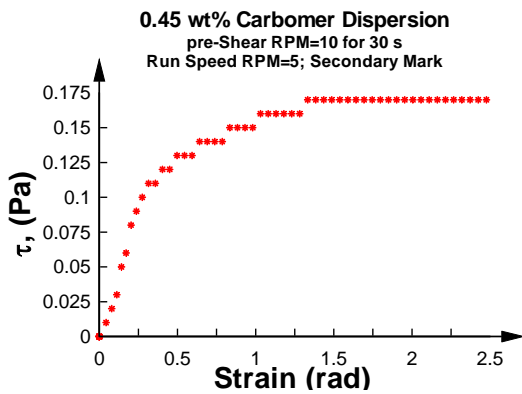


Figure 6: The stress vs. strain for 0.45 wt% carbomer dispersion, pre-shorn for 30 s at RPM=10 and the test was carried out at RPM=5, using spindle LV#71. The yield stress,  $\tau_o$ , with pre-shearing is 0.17 Pa.

Table 3 shows the summary of yield stress tests carried out for carbomer dispersion at different concentrations. One can easily see that the yield stress increases in magnitude with increasing concentration of carbomer, and except with the highest concentration, the yield stress did not change with pre-shearing. At high concentration of carbomer (i.e. 0.9 wt %), the pre-shearing step resulted in a higher value of yield stress; an indication of stiffening or strengthening process (entanglement/build-up of polymer segments) took place. It should be noticed

here that no time was allowed after the step of zeroing the torque reading which is usually done as a priori for the sake of comparison and treating samples equally. Zeroing the torque reading definitely means moving or rotating the shaft clock- or counter-clock wise in a way to diminish the torque reading and is usually carried out at a specified constant speed (i.e., RPM) which of course results in a shearing effect in addition to the pre-shearing step.

Table 3: The yield stress,  $\tau_o$ , value for carbomer dispersion at different concentrations with or without a pre-shearing step. The accuracy measurement for  $\tau_o$  is also shown.

Carbomer Concentration n (wt%)	$\tau_o$ , without pre-shearing, (Pa)	$\tau_o$ , with pre-shearing, (Pa)
0.15	0	0
0.30	0	0
0.45	$0.18\pm 0.009$	$0.17\pm 0.009$
0.60	$0.41\pm 0.019$	$0.41\pm 0.019$
0.90	$0.76\pm 0.019$	$1.39\pm 0.019$

## CONCLUSIONS

It was found that below 0.45 wt % of carbomer content, carbomer dispersion behave as a Newtonian fluid, whereas for a concentration of 0.45 wt% or more, the dispersions start to exhibit a shear-thinning behavior with shear rate and a rheopectic behavior with shear time. Finally, the yield stress results are in harmony with the shear stress shear rate experiments in a way that below 0.45 wt%, a zero-yield stress value was reported and a non-zero value at or above 0.45 wt% of carbomer content. As a result of shearing, the yield stress measured in a pre-shear sample, the



“dynamic yield”, was found to be larger than the yield stress measured for an originally un-disturbed material, the “static head”; an indication of network stiffening or entanglement took place.

#### ACKNOWLEDGMENT

This research was funded by the Dean of Scientific Research at University of Bahrain.

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