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TRANSPORT OF PARTICLE-LADEN FLUIDS THROUGH FIXED-VALVE MICROPUMPS

Ling-Sheng Jang, Christopher J. Morris, Nigel R. Sharma, Ron L. Bardell and Fred K. Forster *

Department of Mechanical Engineering
Campus Box 352600
University of Washington
Seattle, Washington 98195-2600

ABSTRACT

Micropumps designed for the flow-rate range of 100–1000 μl/min have been developed by a number of research groups. However, little data is available regarding the ability of various designs to directly transport liquids containing particles such as cells, microspheres utilized for bead chemistry, or contaminants. In this study the ability of pumps with no-moving-parts valves (NMPV) to transport particles was investigated. The results showed that a NMPV micropump was able to directly pump suspensions of polystyrene microspheres from 3.1 to 20.3 μm in diameter. The pump functioned without clogging at microsphere number densities as high as 9000 particles/μl of suspension, which corresponded to over 90,000 particles per second passing through the pump at a flow rate of 600 μl/min. Performance with polystyrene microspheres was the same as pure water up to the point of cavitation. Microspheres manufactured with negative surface charge cavitated less readily that other microspheres studied that were manufactured without surface charge. However, cavitation did not appear to be a function of microsphere size, total surface area or number density. Thus pumping polystyrene microspheres was found to be more affected by surface effects than by size, surface area or number density within the range of parameters considered. In the case of charged microspheres, the maximum flow rate was reduced by 30% compared to pure water whereas for uncharged microspheres the maximum flow rate was reduced by approximately 80%.

*Address correspondence to this author at the above address or to forster@u.washington.edu.

NOMENCLATURE

C fluid capacitance $(\mathrm{m}^3/\mathrm{Pa})$
V Volume (m^3)
NC Identifies suspensions of microspheres manufactured with no surface charge
WC Identifies suspensions of microspheres manufactured with surface charge
k Gas constant, 1 for isothermal process, ratio of specific heats for adiabatic process
p absolute pressure (Pa)
β Bulk modulus $(\mathrm{N/m}^2)$
μ absolute viscosity (Pas)
ρ mass density $(\mathrm{kg/m}^3)$
ϕ Volume fraction of particles in suspension
$(\ )_c$ pump chamber property
$(\ )_g$ gas property
$(\ )_l$ liquid property
$(\ )_p$ particle property
$(\ )_s$ suspension property

INTRODUCTION

Several designs for micropumps have been developed by different groups based on various principles of actuation and various types of valves. Most pumps are designed to work with pure fluids, and in most cases there is considerable risk of valve clogging if fluid with particles is directly pumped. Micropumps with no-moving-parts valves (NMPV) are of interest because of their simplicity, reliability, ease of manufacture, and potential for being able to pass particles (Forster et al., 1995; Olsson et al., 1995; Bardell et al., 1997). However, there have been no studies on the effect of directly pumping particles through pumps of this design.

Even without complications of particles interfering with the action of valves that open and close, which are minimized in NMPV pumps, particles may affect pump performance indirectly through changes in macroscopic fluid behavior caused by particles. Based on current pump models (Bardell et al., 1997) the fluid properties that affect pump performance are mass density, viscosity and compressibility. In terms of fluidic model parameters these properties are associated with inertance, resistance and capacitance.

Mass density affects fluid inertance, which for a conduit is proportional to the product of density and length and inversely proportional to cross-sectional area. Since the cross-sectional area of the valves is relatively small, valve inertance has significant influence on pump operation. The density of a suspension relative to the liquid phase is given by

$$\rho_s/\rho_l = 1 + (\rho_p/\rho_l - 1)\phi, \tag{1}$$

where ϕ is the volume fraction of particles. For increasing particle mass density, significant relative motions between particles and fluid may develop in NMPV pumps. This behavior may affect the manner in which particles and fluid travel through multi-channel fixed-geometry valves and could alter valve performance.

Fluid resistance in a conduit is proportional to viscosity and a strong function of the minimum transverse dimension. For example, for a conduit of circular cross-section the fluid resistance is inversely proportional to the forth power of diameter. Since the transverse dimension of pump valves is relatively small, valve resistance has a significant influence on pump operation. The viscosity of a dilute suspension characterized by the Einstein relation relates the effective absolute viscosity to that of the liquid and to the volume fraction of particles

$$\mu_s/\mu_l = 1 + 2.5\phi \tag{2}$$

(Probstein, 1989). When the size of particles in a suspension approaches the dimensions of the pump valves, additional viscous effects preclude modeling valve fluidic resistance based on a simple macroscopic change in viscosity. In that case two phase flow and lubrication theory may be required to accurately model valve losses due to viscous effects.

The NMPV micropump operates near resonance by design, and the resonant frequency is strongly dependent on capacitance of the fluid in the pump chamber. At frequencies above resonance a significant amount of membrane displacement that ideally would result in outlet flow through the valves is consumed by the capacitance of fluid in the pump chamber (Bardell et al., 1997). Capacitance arises from fluid bulk modulus, which for a suspension is given by

$$\beta_s/\beta_l = 1/\left[1 - (1 - \beta_l/\beta_p)\phi\right]. \tag{3}$$

The capacitance of a suspension and gas in the pump chamber is given by

$$C_c = V_s/\beta_s + V_g/kp, \tag{4}$$

where $V_c = V_s + V_g$, p is absolute pressure and k is a gas constant that is unity for an isothermal process and the ratio of specific heats for an adiabatic process (Rowell and Wormley, 1997). The capacitance due to gas in the pumped fluid can have a dramatic effect on pump behavior, i.e. the second term in the above equation can be much larger than the first. In addition to the basic manner in which entrained gas affects pump chamber capacitance, the role of particles in entraining such gas may significantly contribute to changes in pump operation.

Due to the many factors described above that have potential effects on pumping suspensions, a series of experiments was conducted to develop a more quantitative understanding of the effects of pumping particle-laden fluids. The experiments included means to investigate many of the factors associated with suspensions. Steady flow tests, frequency analysis and basic pump performance tests were used along with results of an existing system model to investigate the individual factors associated with suspensions.

METHODS

The methods used in this study can be divided into a description of the particular pump utilized, the preparation of the particle suspensions, the test for non-aggregation, steady flow tests, frequency response tests and pump performance measurements.

Pump Description

The design of fixed-valve pump used for all tests is shown in Fig. 1. The pump chamber and valves are shaded darkest. They were etched in silicon to the same depth. An anodically bonded rectangular Pyrex layer was used to seal the valves and chamber. It also acted as a deformable plate where it spanned the pump chamber. Shown shaded lightest and having the smallest diameter is the piezoelectric element. It was bonded to the Pyrex with conductive epoxy to form electrical contact with the bottom surface of the piezoelectric element. The epoxy is shown shaded darker than the smaller piezoelectric element and includes the electric connection region on the upper right side of the pump. The valves connected to plenum chambers where connections to

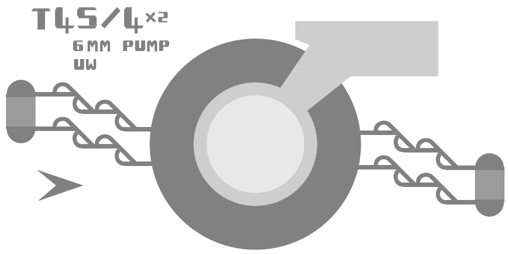

Figure 1. CONFIGURATION OF A T45/4X2 6-MM DIAMETER PUMP. CHAMBER AND VALVES ARE SHOWN DARKEST, PIEZOELECTRIC ELEMENT IS SHOWN LIGHTEST AND CONDUCTIVE EPOXY IS SHOWN IN AN INTERMEDIATE SHADE OF GRAY. THE DIRECTION OF NET FLOW IS INDICATED WITH AN ARROW.

an inlet reservoir and the load were made. Two different micropumps were used. They only differed in the etch depth of the chamber and valves. Deep reactive ion etching (DRIE) was used to etch one pump to a depth of 117 μm and the other to a depth of 156 μm. The pump chamber diameter was 6 mm, and the cover plate was 500 μm thick. The piezoelectric material used for the driving element was PZT-5A (PSI-5A-S2, Piezo Systems, Inc., Cambridge, Massachusetts). It was 5 mm in diameter and 190 μm thick. The transverse dimension of the valves was approximately 114 μm. Further details on the type of fixed-valve pump used have been published previously (Bardell et al., 1997).

Sample Preparation

The suspensions used consisted of two different types of polystyrene microspheres. One type had a nominal diameter (and standard deviation) of 20.3(0.33) μm (PS07N, Bangs Laboratories, Fishers, Indiana) with a specific gravity of 1.062. These microspheres had no special surface treatment and in this study are referred to as NC for not charged. Another type of microsphere used was obtained in three different diameters (and standard deviation) of 3.1(0.11), 4.6(0.19) and 7.9(0.85) μm, respectively (1-3000, 1-4500, 1-8000, Interfacial Dynamics, Corp., Portland, Oregon) with a specific gravity of 1.055. These microspheres were negatively charged with sulfate functional groups on the surface and had a charge density between 5.4 and 5.6 $\mu\mathrm{C/cm}^2$. In this study these microspheres are referred to as WC for with charge.

To prepare suspensions of different microsphere concentration, a known volume of de-ionized water filtered at 0.2 um was combined with a predetermined volume of stock microsphere suspension based on the manufacturers' specification of percent solids. Precision micropipettes (MW128, Chemglass, Vineland, New Jersey) were used to measure the volumes. The diluted WC microsphere suspension was degassed for 10 min with a vacuum pump (N810.3FT8, KNF Neuberger, Inc., Trenton, New Jersey) while the sample was agitated in an ultrasonic bath (Bransonic 12, Branson Instr. Co., Shelton, Connecticut) and kept at room temperature. In the case of the NC microspheres, de-ionized water was degassed as described above, after which the predetermined amount of stock suspension was added. The latter protocol ensured the resulting diluted suspension consisted of non-aggregated microspheres.

The diluted suspensions were introduced into pumps in a manner that completely displaced all air. This was accomplished by first filling the pumps with ethanol.

Aggregation Test

Particle suspensions were checked for aggregation as a part of all pump tests. Before and after each test a small amount of the fluid sample was collected with a syringe labeled for that particle size. An inverted microscope (IM35, Zeiss, Germany) was used in conjunction with a high performance CCD camera (4912-2000/0000, Cohu, Inc. San Diego, California) whose image was displayed on a video monitor. A drop of suspension was placed on a glass slide and viewed through the microscope. A piece of metal foil was used to reflect forward-projected light back through the microspheres to increase their visibility. If more than approximately five percent of the particles in a field of view were aggregated in groups of three or more, the suspension was determined to be aggregated and it was discarded. Otherwise the suspension was determined to be non-aggregated, and the test data was used.

Steady Flow Resistance

A syringe pump (Model 200, KD Scientific, Boston, Massachusetts) was used to provide a known flow rate for steady flow resistance tests on the pumps. A precision syringe (GASTIGHT, Hamilton Company, Nevada) was utilized in the syringe pump. It was connected to one branch of a plastic Y-section from an intravenous filter set (SFE-2017SL, B. Braun Medical Inc., Bethlehem, Pennsylvania) using standard Luer-lock fittings. A pressure transducer (EPI-127, Entran Sensors & Electronics, Fairfield, New Jersey) was connected to the other branch, and the outlet was connected to the pump. The pressure transducer was previously calibrated against a mercury manometer. Flow rates of 1000 to 5500 $\mu\mathrm{l/min}$ were applied, and the DC pressure transducer output was read on an oscilloscope.

Frequency Response Tests

Frequency response of the pump was measured in terms of the peak velocity of the center of the driving piezoelectric ac-

Table 1. CASE DEFINITIONS FOR ALL THE POPULATIONS OF MICROSPHERES USED IN THIS STUDY. WC REFERS TO THE MICROSPHERES MANUFACTURED WITH ADDED NEGATIVE SURFACE CHARGE, AND NC REFERS TO THE MICROSPHERES MANUFACTURED WITHOUT ADDED SURFACE CHARGE. β_p WAS TAKEN TO BE $5 \times 10^9\,\mathrm{N/m^2}$.

case	type	size (μm)	concentration (μg/ml)	number density (beads/ml)$\times 10^{-6}$	surface area (μm^2/ml)$\times 10^{-6}$	$\rho_s/\rho_l - 1$ $\times 10^6$	$\mu_s/\mu_f - 1$ $\times 10^6$	$\beta_s/\beta_l - 1$ $\times 10^6$
A	WC	3.1	70	4.19	126	4	166	37
B	WC	3.1	150	9.22	278	8	356	80
C	WC	4.6	230	4.19	278	12	545	123
D	WC	7.9	390	1.42	278	20	924	208
E	WC	7.9	1100	4.19	821	57	2608	587
F	NC	20.3	250	0.0537	70	15	598	132
G	NC	20.3	1000	0.215	278	58	2354	529

tuator, as a function of frequency at a low driving voltage, and with the pumping system filled with suspension. A vibrometer (Polytec OFV 2600, Germany) was used to measure the velocity. A Sweep/Function Generator (Model 19, Wavetek, United Kingdom) and a piezo amplifier (EPA-102, Piezo system Inc., Cambridge, Massachusetts) were used to drive the piezoelectric actuator with a sinusoidal signal. Twenty-cm long silastic tubes having a diameter of 1 mm (62999-166, VWR Scientific, Brisbane, California) were connected to the inlet and outlet ports of the pump. The inlet and outlet tubes were inserted into open reservoirs, the liquid surfaces of each were at the height of the pump.

Pump Performance Tests

Pump performance tests consisted of measuring the zero load output flow rate and the blocked flow outlet pressure head. For flow measurement the inlet and outlet tubes were configured as described above. A micro "bucket and stop watch" method was utilized. The outlet reservoir was situated on an electronic scale (1205 MP, Sartorius, Westbury, N.Y.), and change in weight over a know time interval was used to calculate flow rate. The blocked flow pressure was measured with the pressure transducer that was used for the steady flow tests connected to the end of the outlet tube.

RESULTS AND DISCUSSION

Seven different suspensions were considered from four different sized microspheres. Table 1 summarizes the factors that differentiated each suspension. In the table values for relative density, viscosity and bulk modulus were calculated with Eqs. 1, 2 and 3. Four cases were at concentrations that yielded the same surface area but with distinct sizes, and three cases were at concentrations that yielded the same number density but also with distinct sizes. Cases of equal surface area and number density were chosen to highlight any effects that might correlate with either parameter.

To perform all tests each suspension was pumped for a minimum of 30 minutes. During no test was any clogging of a pump observed. For a given number density and flow rate, the actual number of particles successfully pumped per unit time was substantial. For example, when suspension B was pumped at a flow rate of 600 μl/min, a pumping rate of 5.4 million particles per minute was attained.

Examples of suspensions with and without aggregation are shown in Figs. 2 and 3. From such micrographs, it was easy to assess whether a suspension aggregated. Aggregation was a problem with the NC microsphere suspensions, which were degassed using a modified procedure due to their tendency to aggregate. The NC microsphere suspensions eventually aggregated over time after preparation, but with the method used to monitor aggregation, pump data was easy to categorize in that respect. All data reported below corresponded to suspensions that did not aggregate and therefore corresponded to well-defined microsphere sizes.

The purpose of measuring pressure drop across a non-operating pump as function of steady flow rate was to investigate the role of suspension parameters on viscous effects associated with the NMPV pump. Since the flow was steady, it was assumed that any effects of inertance or capacitance were negligible compared to the case of oscillating flow, i.e. during pump operation. The pressure drop for pure water and various suspensions is shown in Fig. 4 as a function of volume flow rate in both the forward and reverse directions. The resistance is given by the

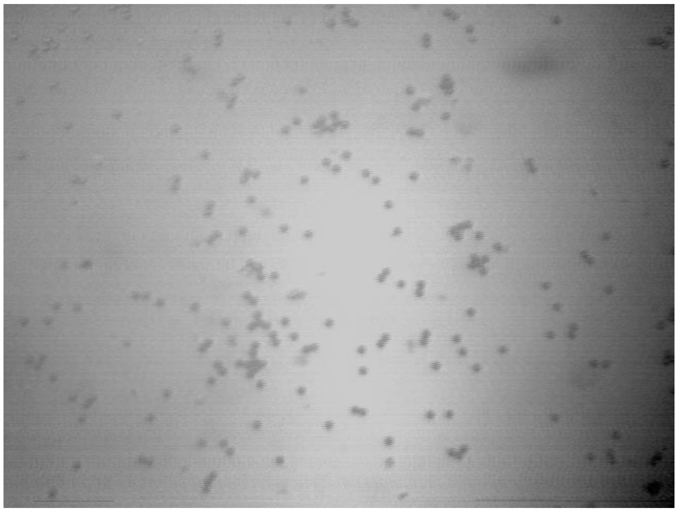

Figure 2. AGGREGATED 20.3 μm NC MICROSPHERES IN A DROPLET OF SUSPENSION, CASE G IN TABLE 1, RECORDED WITH A CCD CAMERA AND A 4X OBJECTIVE LENS.

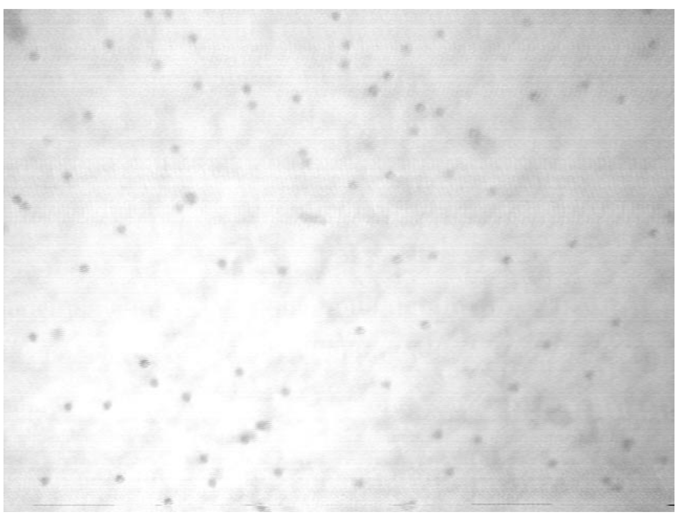

Figure 3. NON-AGGREGATED 7.9 μm WC MICROSPHERES IN A DROPLET OF SUSPENSION, CASE D IN TABLE 1, RECORDED WITH A CCD CAMERA AND A 10X OBJECTIVE LENS.

local slope of the pressure versus flow curve. There was very little difference between the resistance of pure water, and the resistance of any of the particle suspensions. From the data presented it can be concluded that viscous effects due to the particle parameters were negligible. Since Eq. 2 predicts a very small effect on the effective viscosity of any of the suspensions utilized, the data supports that prediction. Less obvious, however, is that even in the case where the valve minimum dimension to particle size ra-

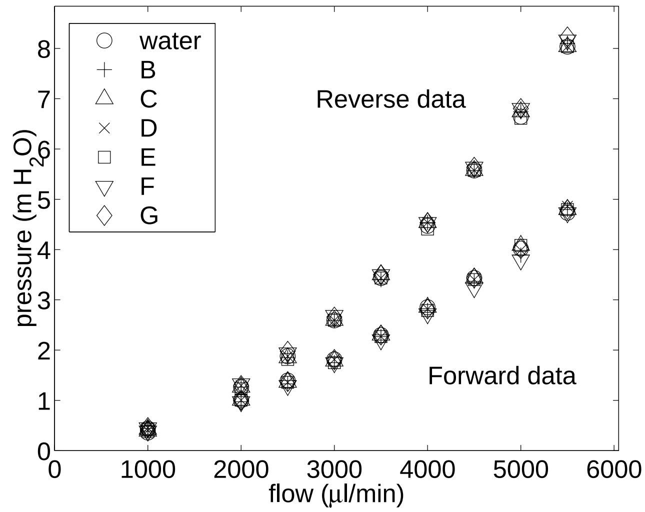

Figure 4. PRESSURE DROP VERSUS STEADY FLOW RATE FOR PURE WATER AND VARIOUS PARTICLE SUSPENSIONS DESCRIBED IN TABLE 1 AND THROUGH THE PUMP IN FIG. 1.

tio was approximately six, no detectable effects were seen in the resistance values in forward or reverse flow. Thus, the current NMPV pump is not influenced by viscous flow effects caused by particle sizes as large as 20 μm for the concentrations considered. This is very encouraging because particles of that size are larger than the valve clearance of many micropumps designed with moving valve geometry.

The pump used for the above steady-flow tests was 25% shallower than the pump used for all the other tests. Since no difference in flow resistance between any suspension and pure water was detected for that pump, no difference would be expected for the deeper pump used for the rest of the tests. It should also be noted that for all steady flow resistance results, reverse flow data exhibited higher resistance than the forward flow data. This was expected, because this difference allows the valves to generate a net flow in the direction of smaller resistance when subjected to an oscillating pressure. This characteristic of fixed-geometry valves allows them to function as pump valves.

With the knowledge from the steady-flow tests that viscous effects on pumping are unchanged by any of the parameters of the suspensions considered, the dynamic response of the pump was used to investigate the effect of microspheres on fluid capacitance. Figure 5 is a plot of the frequency dependent behavior of the pump around the resonant frequency associated with pumping, i.e. a resonance that is related to fluid flow in the valves and dependent on the fluid resistance and inertance in the valves and the capacitance of fluid in the pump chamber (Bardell et al., 1997). The response shown in the figure is the centerline velocity of the membrane. Other output parameters such as valve flow

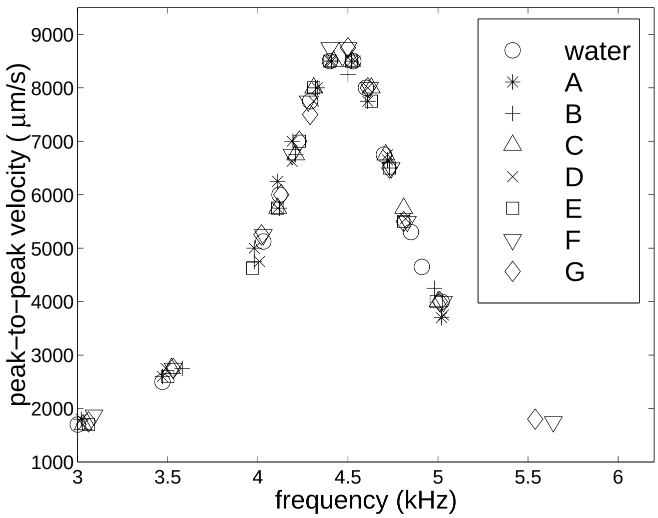

Figure 5. MEMBRANE PEAK CENTERLINE VELOCITY VERSUS FREQUENCY AT 15V PEAK-TO-PEAK FOR PURE WATER AND VARIOUS PARTICLE SUSPENSIONS DESCRIBED IN TABLE 1.

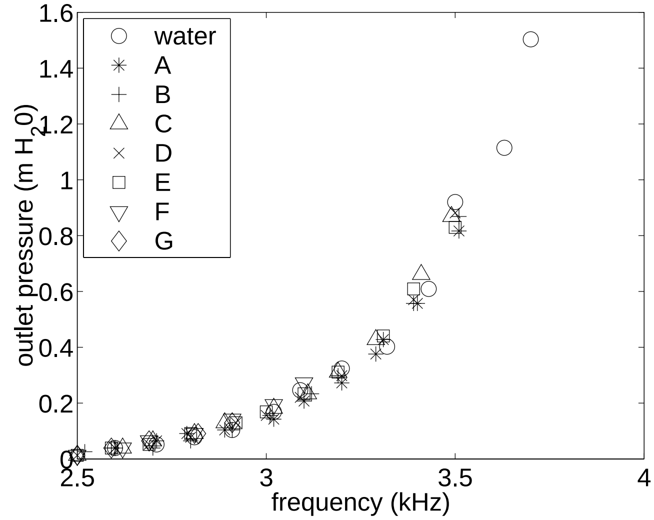

Figure 7. BLOCK LOAD OUTLET PRESSURE AS A FUNCTION OF FREQUENCY AND A 170 V PEAK-TO-PEAK DRIVING SIGNAL FOR PURE WATER AND VARIOUS PARTICLE SUSPENSIONS DESCRIBED IN TABLE 1.

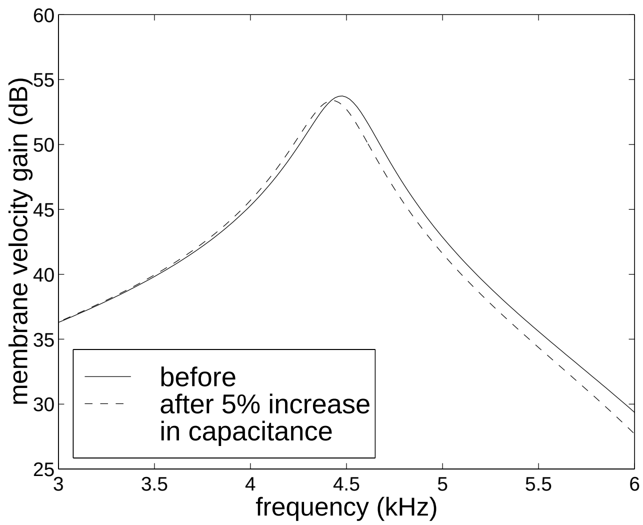

Figure 6. CALCULATED EFFECT OF CHAMBER CAPACITANCE ON THE FREQUENCY RESPONSE OF PUMP MEMBRANE CENTERLINE VELOCITY. THE INPUT PARAMETERS CORRESPOND TO THE PUMP SHOWN IN FIG. 1. ZERO dB $= 1\mu$m/s AT A 1V PEAK-TO-PEAK DRIVING VOLTAGE.

and chamber pressure would show similar behavior. It is clear that there is no discernable difference in magnitude or frequency of the peak response. However, the results of a linear model (Bardell et al., 1997) of the pump in Fig. 6, show that with just a five percent change in the amount of entrained gas in the chamber, the resonant frequency shifts noticeably. It is possible that a similar variation in valve inertance could cause a corresponding change in resonant frequency, but the changes in density due to particles shown in Table 1 are less than 0.01%. Thus the capacitance effects on pump operation were unchanged by any of the parameters of the suspensions considered.

Having found that inertance, resistance and capacitance effects of the various suspensions did not cause any observable effects on pump characteristics, one would expect that the pump performance would be unaffected by any of the suspensions considered compared to water. Figure 7 shows that to be the case in terms of the block load pressure versus frequency for a typical driving signal of 170 V peak-to-peak up to a frequency of approximately 3000 Hz, after which both NC suspensions cavitated. To look more closely at any possible trend in performance for a frequency at which all suspensions were pumped data from Fig. 7 at 2.9 kHz and similar data for no-load flow rate were utilized to generate the pump performance curve shown in Fig. 8. The lines drawn between data points are typical of performance obtained with more data (Bardell et al., 1997). However, the figure shows no correlation between performance and microsphere size, surface area or number density.

A very different result was obtained when the *maximum* performance attainable with each suspension was compared as shown in Fig. 9. The maximum performance was determined with an input signal of 170 V peak-to-peak and by increasing the frequency of the input signal until cavitation occurred. Clearly the results grouped into three distinct regions corresponding to

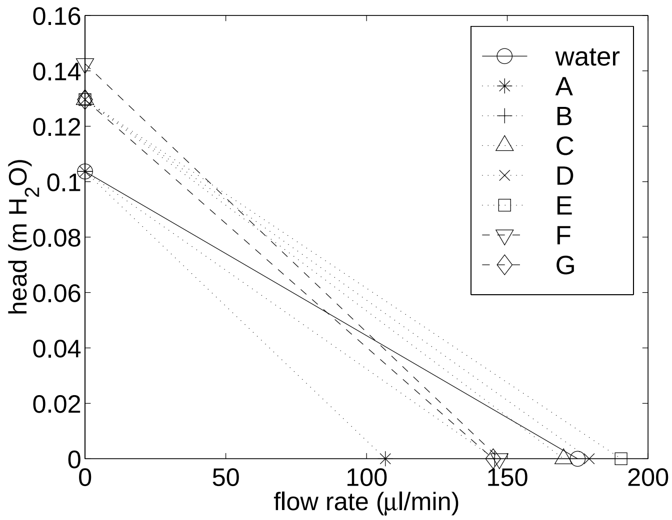

Figure 8. PUMP PERFORMANCE CURVES AT A TYPICAL OPERATING POINT OF 2900 HZ AND 170 V PEAK-TO-PEAK DRIVING SIGNAL, FOR PURE WATER AND VARIOUS PARTICLE SUSPENSIONS DESCRIBED IN TABLE 1. LINES DRAWN FOR CLARITY.

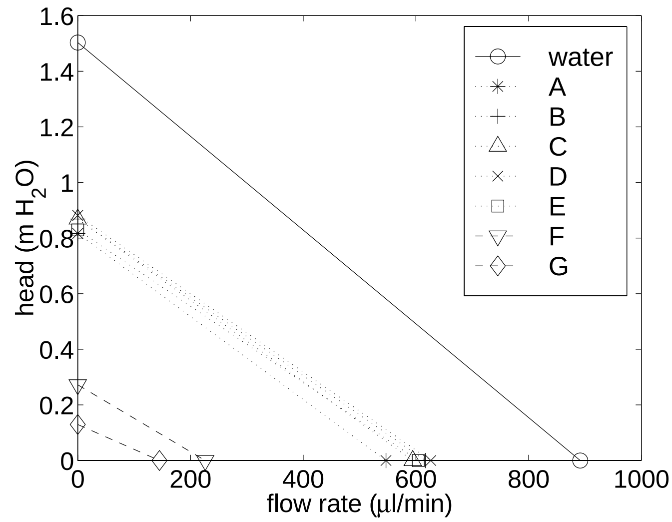

Figure 9. MAXIMUM PUMP PERFORMANCE ATTAINED BEFORE CAVITATION WITH 170 V PEAK-TO-PEAK DRIVING SIGNAL FOR PURE WATER VARIOUS AND PARTICLE SUSPENSIONS DESCRIBED IN TABLE 1. EACH CASE CORRESPONDED TO THE DRIVING SIGNAL FREQUENCY THAT YIELDED THE HIGHEST PERFORMANCE. LINES DRAWN FOR CLARITY.

pure water (having the highest performance), then the WC suspensions and finally the NC suspensions. This grouping is also evident in Fig. 7 where the maximum NC frequency is approximately 3 kHz, where the maximum WC frequency is 3.5 kHz and where the maximum frequency before cavitation occurred for pure water is 3.7 kHz. Thus, the primary parameter that produced variations in pumping characteristics was the tendency to cavitate. Both WC and NC microspheres were polystyrene and hydrophobic according to the manufacturers. However the WC negatively charged particles were significantly more resistant to cavitation. We conclude that the tendency for particle suspensions to cavitate while being pumped is significantly affected by surface chemistry. This effect was significant enough to override a seven-fold difference in microsphere diameter, an 80-fold difference in number density and a 12-fold difference in surface area.

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