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Date June 9 1982

A MODEL SYSTEM FOR THE STUDY OF PARTICLE DETACHMENT KINETICS IN FLUIDIZED BEDS

by

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ABSTRACT

The concept of fluidized bed as applied to backwashing of filters was used to study particle detachment of uniform spherical size alum sol particles of narrow size distribution, from glass spheres. The optimum backwashing theory developed by Amirtharajah [1] was used to analyze the system. A first order rate expression fitted the data well. Even under optimum conditions of backwashing, a significant fraction of attached particles could not be separated. This is in good agreement with the mathematical expressions developed by Amirtharajah and Giourgas [3]. The heterogeneity of the surface, as revealed by microscopic examinations, might significantly influence particle detachment. The model system used confirmed some of the accepted facts in backwashing. The chosen system meets several requirements for a successful analysis and thus makes itself available for particle detachment studies.

INTRODUCTION

The fluidized bed is a solid-liquid contact system where the solid particles are suspended in an upward flowing liquid stream and the motion of the particles distinguishes it from other processes (fixed beds). This technique finds its application in various water and wastewater treatment processes, one of which is backwashing of filters. Backwashing is a process where the filter is washed by reversing the flow of water through the filter at a rate so as to cause the expansion of the filter medium. The deposited material is thus flushed through the expanded bed and wasted. There is little fundamental understanding of the particle detachment from the suspended solid phase which occurs during backwashing of filters.

Amirtharajah [1] established that particle collision and abrasion in a fluidized bed is limited, and that hydrodynamic forces are the principal cleaning mechanism in backwashing filter beds. In recent years, Matijevic and coworkers [6,7] have been involved in the studies of particle adhesion and removal in model systems, using monodispersed chromium hydroxide and hematite particles prepared by hydrothermal aging. In their experiments they simulated a fixed bed system with laminar flow (low Reynolds number) and the desorption phenomena was interpreted in terms of classical double layer theory and the diffusional escape of the particles across the interaction energy barrier [7].

The flow regime in a backwashed filter is in the transitional state. The fluid/particle system creates continuously fluctuating velocities around a macroscopic mean velocity for both particles and fluid. Thus, a theoretical model based on turbulent conditions can be developed. This approach was adopted by Amirtharajah and Giourgas [3] to develop a new theory for particle detachment from sand grains. The theory predicts that particulate fluid-

ization is not a completely effective cleaning method, and possibility of deposit buildup at the end of each backwash operation exists. Matijevic and coworkers [6] observed that complete removal of particles could not be obtained in the systems they studied.

Adequate analysis of the kinetic data might render better understanding of the detachment mechanisms and a well defined system will aid in this process. The objective of this work was to develop one such system and test it with some of the existing backwashing theory [1,3].

THEORETICAL CONSIDERATIONS

Amirtharajah's method [10] is used for the estimation of desired wash rates (gpm/ft^2) for various bed expansions. The minimum fluidization velocity, V_f, is calculated from the empirical nonhomogenous Equation (1):

$$V_f(\text{gpm/ft}^2) = \frac{0.00381\,(d_{60\%})^{1.82}\; W_s(W_m - W_s)^{0.94}}{\mu^{0.88}} \qquad (1)$$

where

$d_{60\%}$ = 60% finer size of the sand, mm = effective size × uniformity coefficient

W_m = specific weight of the sand, lb/ft^3

W_s = specific weight of the water, lb/ft^3

μ = water viscosity, centipoise

The dimensionless Reynold's Number, $Re_f = \rho_e V_f d_{60\%}/\mu$ corresponding to a minimum fluidization velocity and the 60% finer sand size is then calculated. If Re_f is greater than 10, a multiplying correction factor K_R, must be applied to the above value of V_f given by

$$K_R = 1.775 \times Re^{-0.272} \qquad (2)$$

The unhindered settling velocity, V_s, of the hypothetical average particle is then calculated from the following expression:

$$V_s = 8.45\, V_f \qquad (3)$$

The Reynolds number for this particle, based on the unhindered settling velocity conditions, $Re_o = \rho_e V_s d_{60\%}/\mu$ is then determined for use in calculating the expansion coefficient, n_e, which is given by

$$n_e = 4.45\ Re_0^{-0.1} \tag{4}$$

This value of n_e is then used to find the constant K_e for the particular system, using the values of velocity and porosity at minimum fluidization, V_f and ϵ (porosity of the unexpanded bed):

$$V = K_e (\bar{\epsilon})^{n_e} \tag{5}$$

where

V = superficial flow velocity of the water above the sand

$\bar{\epsilon}$ = porosity of the expanded bed

Using the value of K_e, as determined from the preceding equation, expanded porosity at any desired face velocity V can be calculated.

The expanded bed depth is calculated from

$$\frac{D_e}{D} = \frac{(1 - \epsilon)}{(1 - \bar{\epsilon})} \tag{6}$$

where

D = depth of unexpanded bed

D_e = depth of expanded bed

The above mentioned equations were used for calculating the desired expansion and the corresponding backwash water rates (gpd/ft^2) for glass beads 100 μm in diameter.

Amirtharajah [1] has developed a theory for estimating the optimum hydrodynamic shear stress in a fluidized bed. The liquid field was treated as turbulent and the total stress τ included the viscous stress and the Reynold's turbulent stress giving

$$\tau = (\mu + \eta)\left[\frac{gk\epsilon^{(n-1)}}{\nu}\frac{(\rho_s - \rho_f)(1 - \epsilon)}{\rho_f}\right]^{1/2} \tag{7}$$

in which η = Boussinesq's eddy viscosity, k = constant for a system = V_T (terminal settling velocity) for a fluidized bed with equisized particles, ρ_s = density of fluidized solids, g = acceleration due to gravity,

τ can be expressed in the form,

$$\tau^2 = \beta[\epsilon^{n-1} - \epsilon^n) \tag{8}$$

in which β = constant for a system.

Differentiating Eqn. (8)

$$2\tau \frac{d\tau}{d\epsilon} = \beta[(n-1)\epsilon^{(n-2)} - n\epsilon^{(n-1)}] \tag{9}$$

$$= \beta \epsilon^{(n-2)}[(n-1) - n\epsilon] \tag{10}$$

The maximum shear stress is given by

$$\frac{d\tau}{d\epsilon} = 0, \text{ therefore,}$$

$$\epsilon_{opt} = \frac{(n-1)}{n} \tag{11}$$

Applying the conditions used for sand, Amirtharajah [1] determined that maximum shear stress and optimum cleaning should occur at a porosity close to 0.68.

Amirtharajah and Giourgas [3] deduced that the lift force is greater than the adhesive London-Van der Waals' forces only up to a certain distance and subsequent analysis determined that complete removal of particles during backwashing of filters is not possible.

Particle detachment data obtained in the experiments conducted was analyzed using first order kinetic expression similar to the one by Kallay and Matijevic [6]. Recently a new model was developed by Amirtharajah [2] to determine the backwash water quality. The final result of the theory is Equation (12).

$$C = K e^{-(V_x t/d)} \tag{12}$$

where C = concentration, V_x = superficial velocity, d = diameter of the collector and K = $\bar{m}A_o/V$, where $\bar{m}$ = time averaged mass transfer flux, A_o = initial area and V = an arbitrary volume of liquid.

The equation will not be valid at extreme values of t, i.e., as $t \rightarrow 0$ or ∞. Transforming the equation by taking logs,

$$\ln C = \ln K - \left(\frac{V_x t}{d}\right) \tag{13}$$

Thus a plot of ln C versus ($V_x t$) would show a linear variation with a -Ve slope. This is analogous to a first-order rate expression. The Backwash Water Quality Model developed by Amirtharajah [2] provides a rational explanation for some accepted backwashing concepts.

MATERIALS AND METHODS

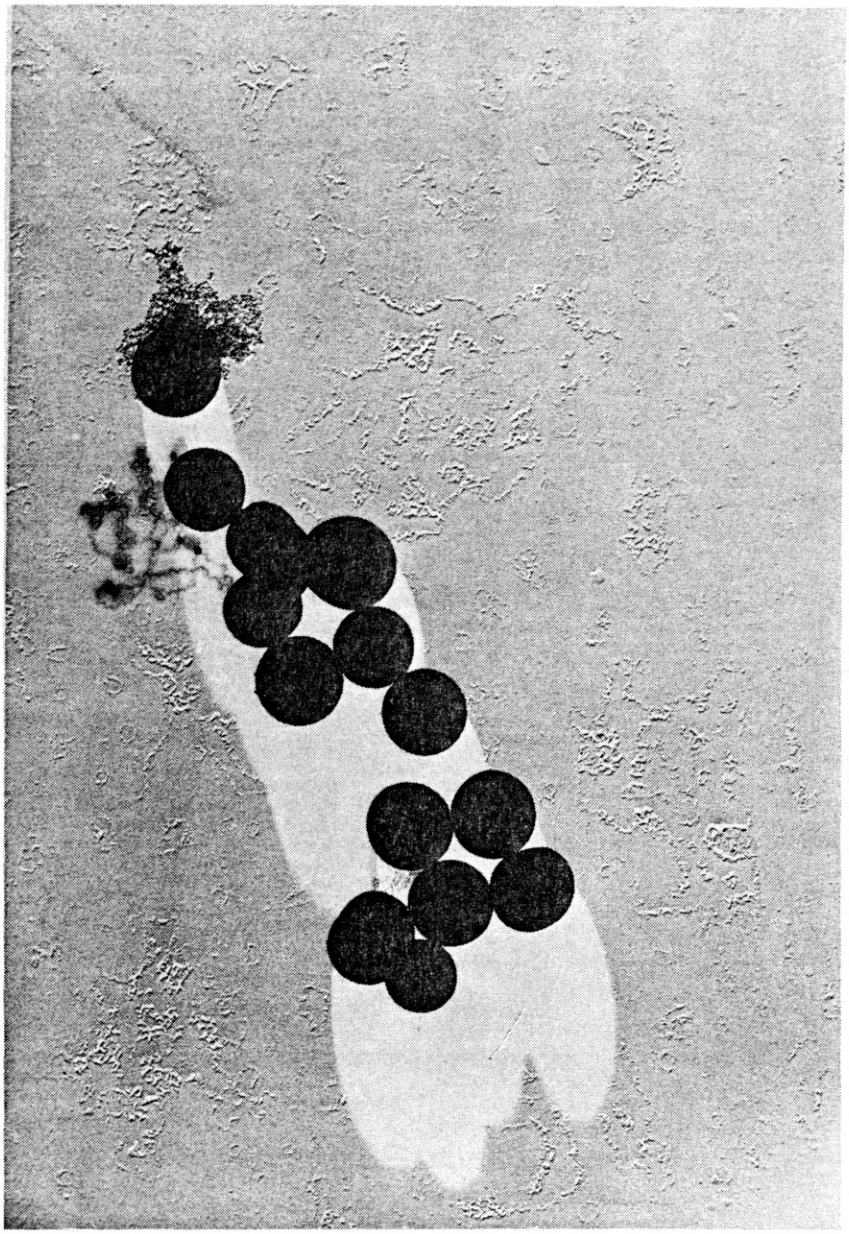
The experimental procedure involved the preparation and stabilization of alum sol particles and subsequent use of these uniformly distributed spherical particles to study particle detachment.

The alum sol preparation essentially consisted of aging at elevated temperatures for 24 hours, solutions of potassium aluminum sulfate containing sulfate as the complexing ion. The procedure was developed by Brace and Matijevic [4]. The sol is stabilized to prevent the dissolution of particles and consequently enhancing the lifetime of particles. Stabilization was carried out by raising the pH of the sol solution to 9.7 at the completion of the aging period. Further purification of the stabilized particles was ensured by centrifugation and subsequent redispersion in deionized water.

The stock solutions used were prepared from "Baker Analyzed Reagents" without further purification. Deionized water was used for all these preparations. This was important in order to prevent heterogenous nucleation during sol formation. All pyrex glassware used were detergent cleaned, acid washed and dried. The alum sol preparation conditions are presented in Table 1.

A fairly large volume of $KAl(SO_4)_2 \cdot 12H_2O$ solution was used to get a concentrated suspension of the sol particles. This was diluted after purification to get a reasonable turbidity, and thus minimize errors in light scattering analysis [8]. The pH of the sol, prior to its attachment on the glass bead, was adjusted to 7.35 ± 0.05, by either dilute acid (HNO_3) or base (NaOH).

The experimental set up used for particle detachment studies is shown in Figure 2. The alum sol solution contained in a one liter Erlenmeyer flask was filtered through a



Size 1 micron = 13.5 mm

Figure 1. Transmission Electron Micrograph of 1×10^{-3} M $KAl(SO_4)_2 \cdot 12H_2O$ After 24 Hours of Aging at 98°C.

Table 1. Alum Sol Preparation Conditions.

Parameters	Reagent Used	
	$Al_2(SO_4)_3 \cdot 16H_2O$ (M.W. 630-39) 2×10^{-3} M as [Al]	$KAl(SO_4)_2 \cdot 12H_2O$ (M.W. 474-39) 1×10^{-3} M as [Al]
Time of aging (hrs)	94	94
Temperature of aging °C	98 ± 2	98 ± 2
pH		
initial	4.1	4.2
final	2.95 ± 0.1	2.9 ± 0.1
Particle size* obtained (micron)	0.9 ± 0.1	0.9 ± 0.1†

*Stabilization with NaOH exchanges sulfates for hydroxyls of the base causing a shrinkage of particle diameter by 10%.

†Figure 1 shows a Transmission Electron Micrograph of the alum sols obtained for 10^{-3} M $KAl(SO_4)_2 \cdot 12H_2O$ after 24 hrs. These sols were used in the subsequent operations due to the shorter aging time.

pyrex glass column (1 inch diameter and 30 inches high), having 100 micron glass spheres for filter medium. The filtration rate was regulated to around 1 gpm/ft^2, with a flowmeter, the main aim was to get the particles attached to the glass beads. The filtered effluent was recycled until the final effluent had a very low and constant turbidity. At this stage, it was presumed that all the particles contained in the alum sol solution had been attached onto the filter media.

The detachment of the particles was carried out, using deionized water. The clean backwash water was contained in a 20-liter glass reservoir and pumped into the column. The flow rate of the clean backwash water was regulated with a flowmeter. The temperature of the backwash water in all the runs remained steady around 20 ± 1°C.

The backwash water carrying the detached particles was collected at regular interval of time and stored in polythene bottles. At the end of each backwash run, the samples

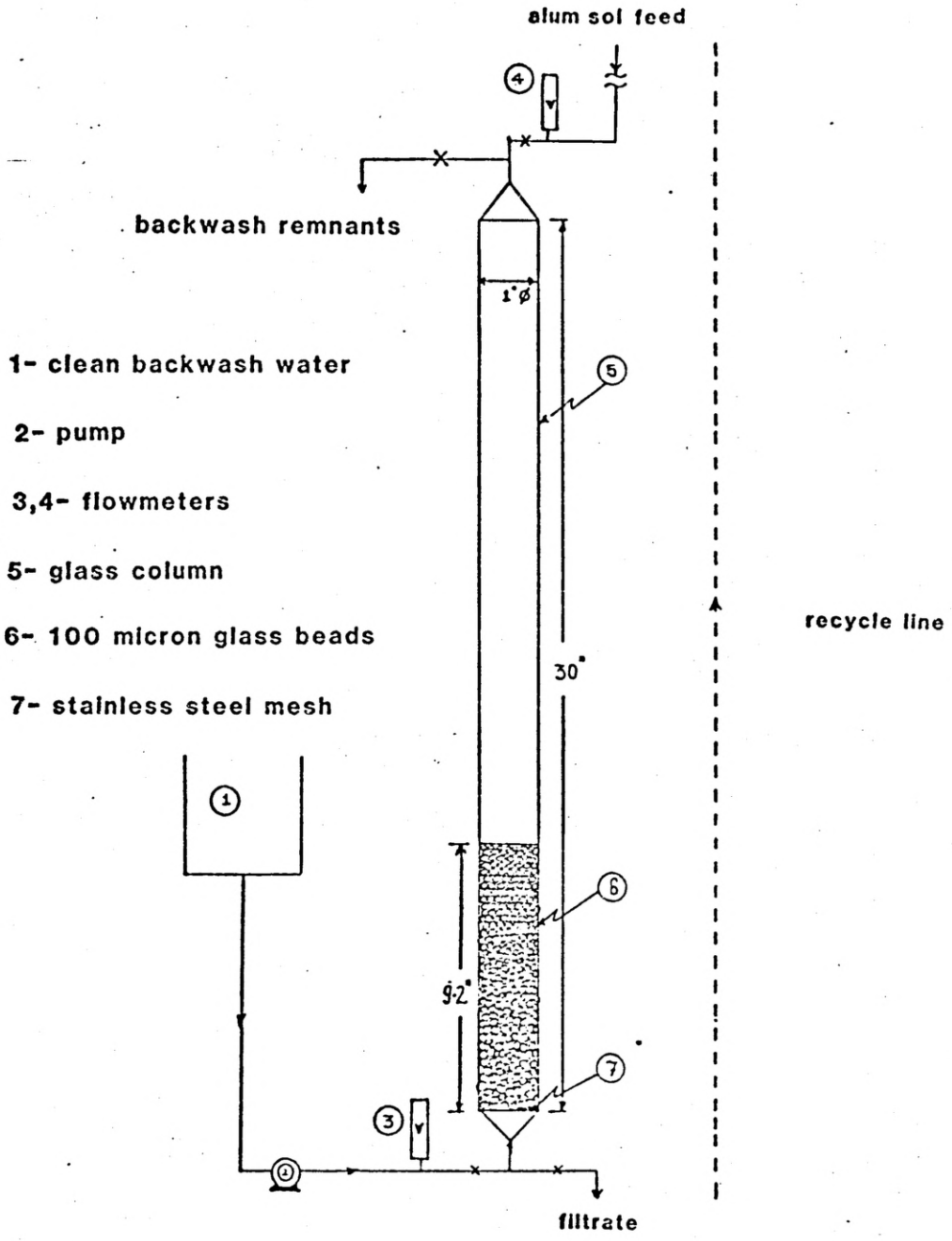


Figure 2. Experimental Set Up.

were analyzed. All the sample bottles and glassware were detergent cleaned and acid washed. The lines carrying the alum sol and the glass column were rinsed thoroughly, prior to each run. The filter medium (glass spheres) was replaced for each new run.

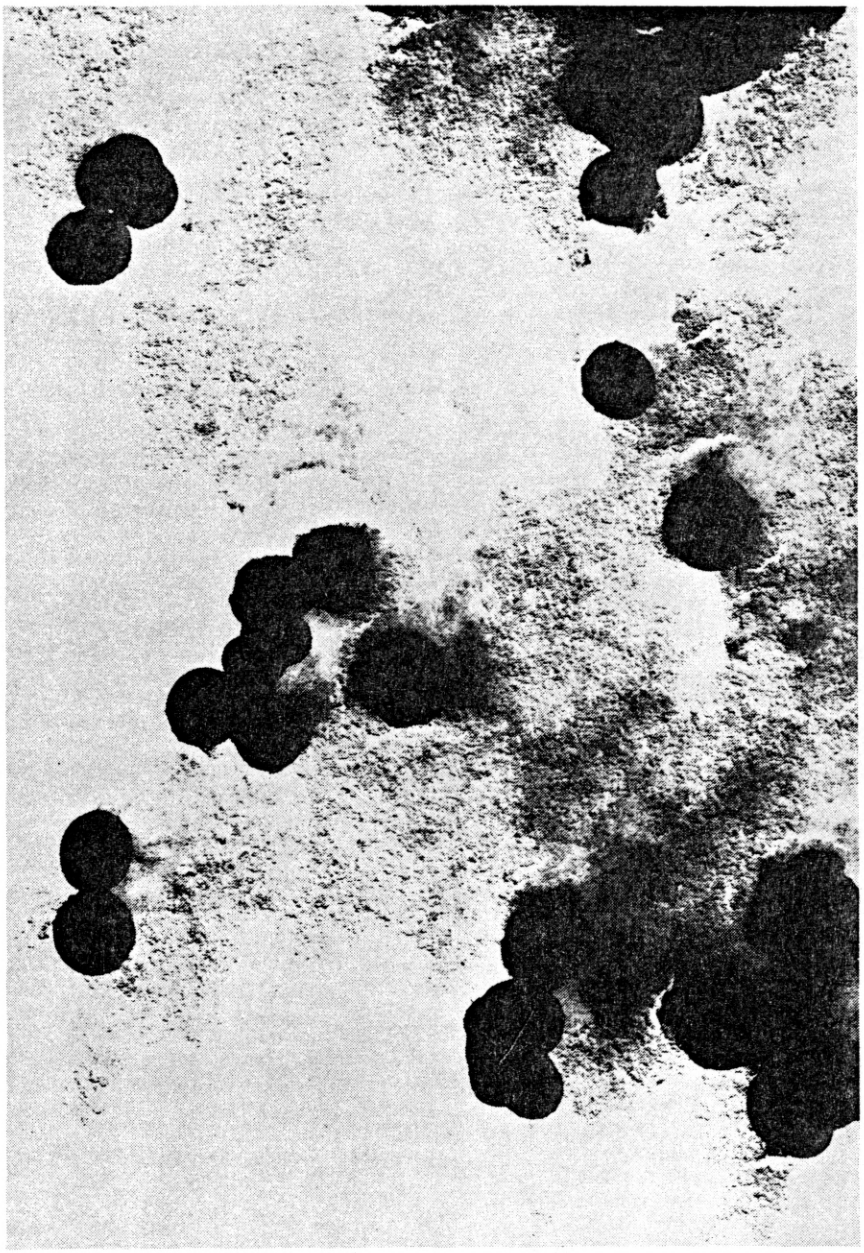
The following analyses were carried out:

Particle Size and Number

A Carl-Zeiss Transmission Electron Microscope was used to determine the particle size and shape. The procedure involved centrifugation of the alum sol sample and placing it on a parlodian coated copper and excess moisture was removed by vacuum evaporation and the resulting sample was viewed under the microscope. The dirty backwash water, through the column, carrying the detached particles was analyzed in a similar manner. A typical electron micrograph from one of the runs is presented in Figure 3.

For the determination of particle number, a known volume of the stock sol solution prior to a run was placed overnight in an oven at 98°C and the dry weight measured. The density of the alum sol was assumed to be 2.42 gm/cc [8]. This is a typical value for hydrous aluminum oxide [5]. Knowing the density, dry weight and the size of the particle, a number concentration of particles was determined. The particle number concentration was correlated with turbidimetric measurements which was carried out with a HACH 2100 Turbidimeter. An appropriate calibration curve was developed (Fig. 4). Thus the turbidity of the backwash remnants could be correlated to the particle number concentration.

Sulfate analysis. The procedure outlined by Vogel [9] was used. A calibration curve was generated using blank samples of known sulfate concentration (Fig. 5). Sulfate analysis



1 micron = 13.5 mm

Figure 3. Electron Micrograph of Alum Sol Particles in the Dirty Backwash Water.

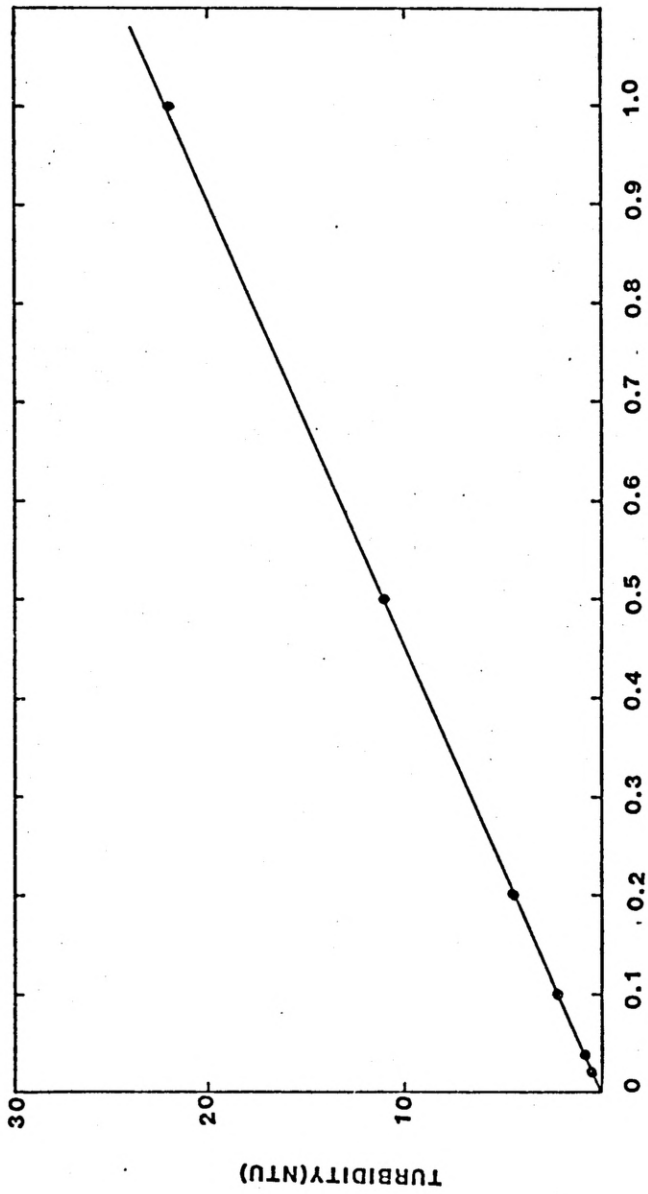


Figure 4. Turbidity Calibration Curve

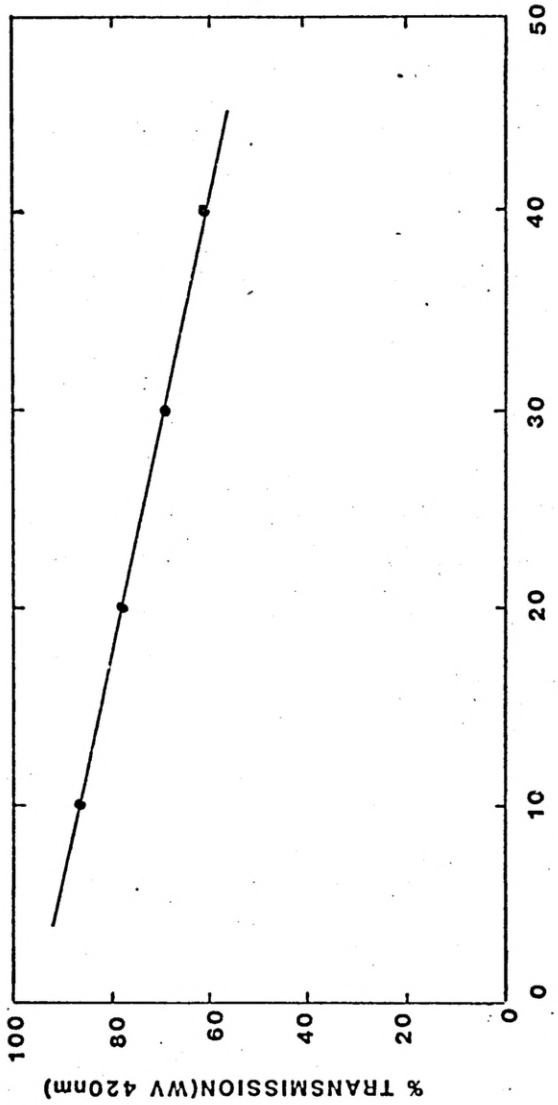


Figure 5. Sulfate Calibration Curve

was done on the alum sol feed, and was found to be negligible. Thus indicating that the sulfate ions were completely replaced by hydroxyl ions from NaOH.

Electrophoretic mobility was determined using the zeta meter and a Van Gill cell. At 10-20 particles were followed and an average time to travel one standard micrometer division was calculated. A standard calibration curve provided by the manufacturers was then used to determine the electrophoretic mobility. Measurements were done on alum sol prior to attachment and also on the detached particles (backwash remnants).

pH measurements. pH measurements were done on alum sol and the backwash water coming through the column using a combination glass electrode and 801 A Orion ionalyzer.

Determination of Number of Particles

Initial turbidity of alum sol = 22.0 NTU

Dry weight of alum sol = 146 mg/l

Density of alum sol = 2.42 g/cc

Volume of one particle = 5.2359×10^{-13} cc (where particle diameter = 1 micron)

Weight of each particle = 1.26×10^{-9} mg/particle

Total no. of particles = 1.15×10^{8} per ml.

Making a series of dilutions, the turbidity of the alum sol was related to particle number. This curve (Fig. 4) was used to determine particle number in the backwash water remnants at the end of each run.

Table 2. Experimental Conditions

Parameters	Alum Sol	Clean Backwash Water	Dirty Backwash Water
pH	7.35 ± 0.05	6.0 ± 0.1	6.2 ± 0.1
Turbidity (NTU)	–	0.1	
Total Dissolved Solids (mg/L)	–	2.5	–
Temperature	–	20.0 ± 1°C	20.0 ± 1°C
Electrophoretic Mobility μm/sec per V/ per μm	-0.6 ± 0.1	–	1.2 ± 0.2

RESULTS

Figure 6 shows the removal of adhered alum sol particles from glass beads on backwashing of the filter at different bed expansion. The fraction of particles removed is obtained by dividing the number of particles detached by the total number of alum sol particles initially attached.

Figure 7 is a plot of fraction of particles still attached on the glass beads at various intervals of time, during backwashing. Figure 8 is a first order kinetics plot for the data obtained for 50% bed expansion. Data points are plotted for three runs under identical conditions.

Figure 9 plots the cumulative removal versus expanded bed porosity as calculated using Amirtharajah's method [10].

Scanning Electron Micrographs (Fig. 10) for 100% expansion were taken to determine surface characteristics, of the glass bead, during the various stages of the experimental run.

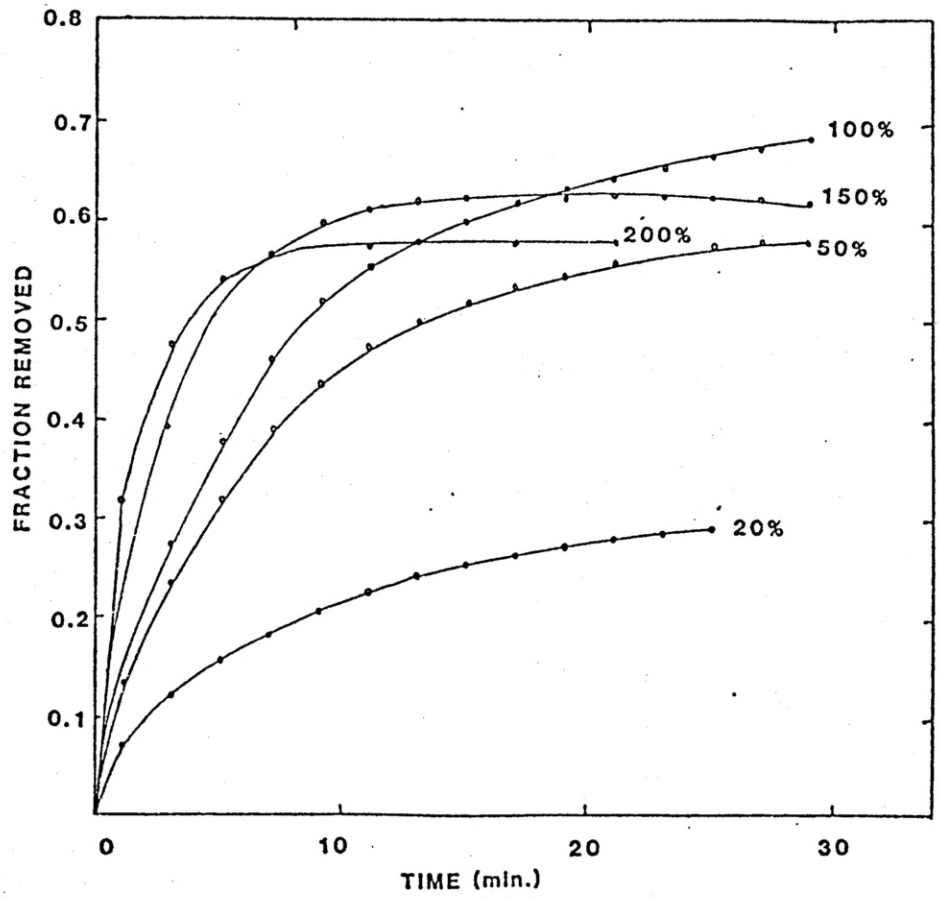


Figure 6. Fraction Removed versus Time.

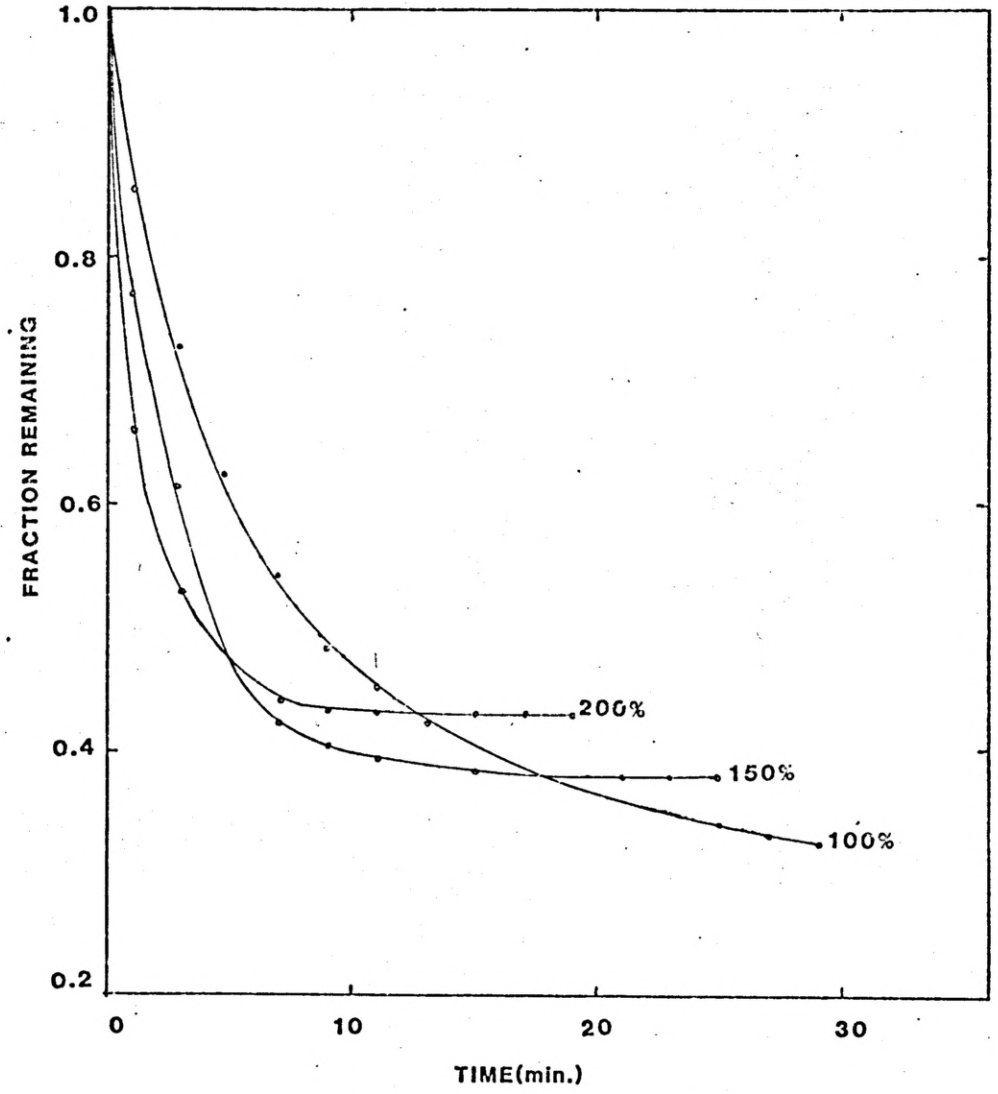


Figure 7. Fraction Remaining versus Time.

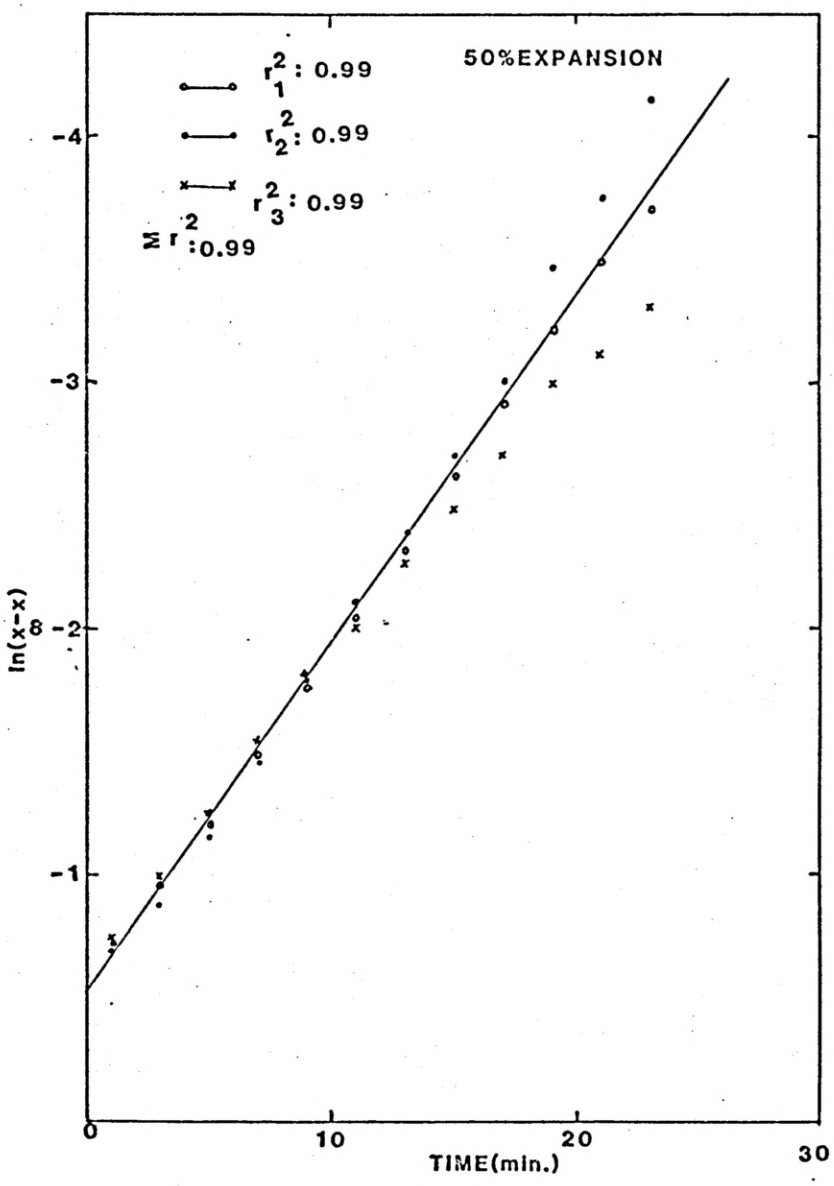


Figure 8. First Order Kinetic Plot For 50% Expansion.

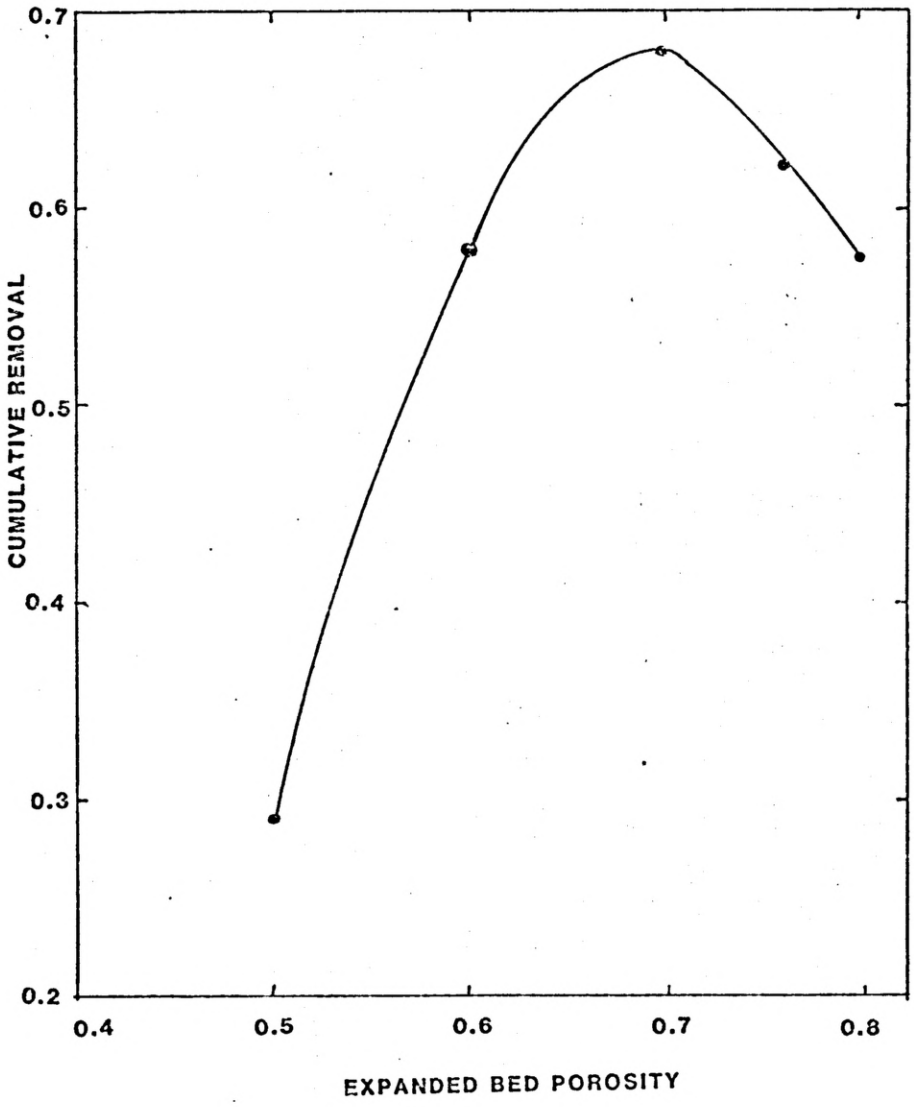
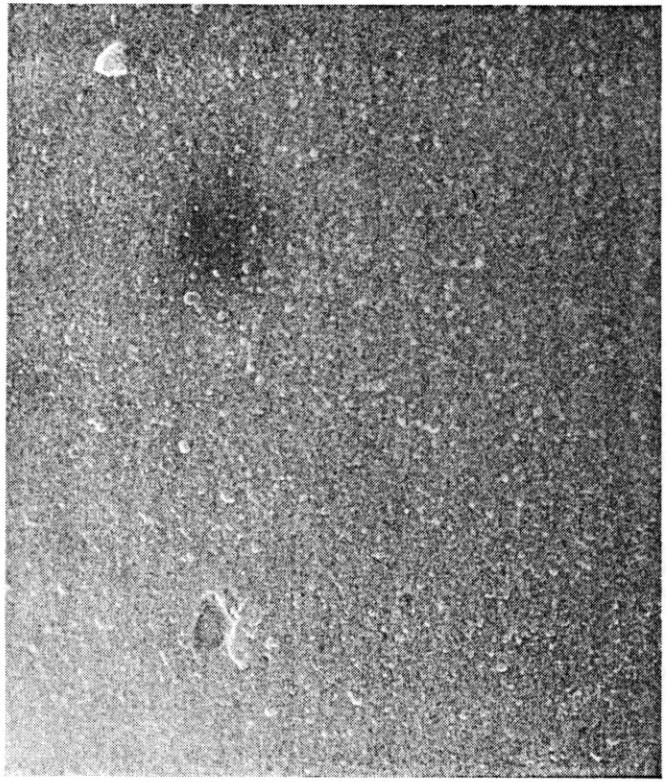
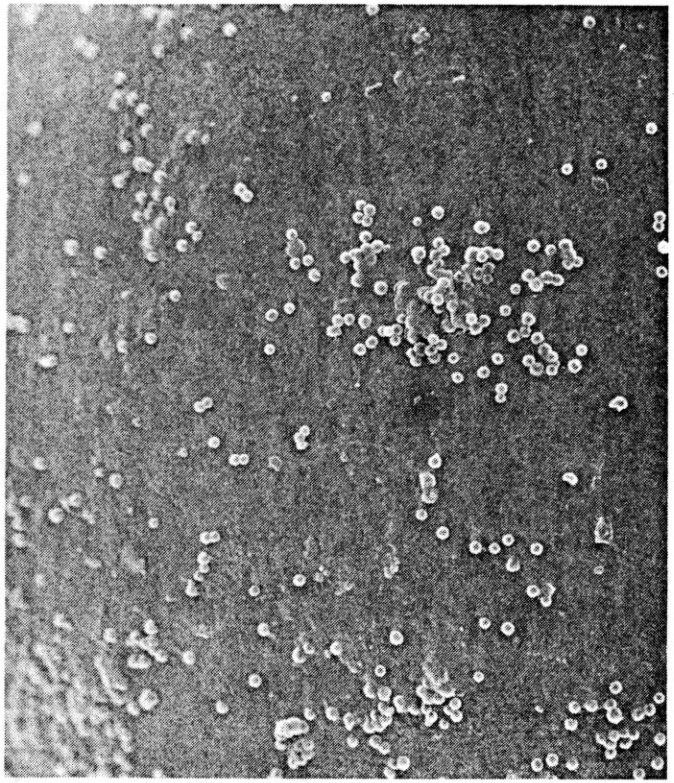


Figure 9. Cumulative Removal versus Experimental Bed Porosity.



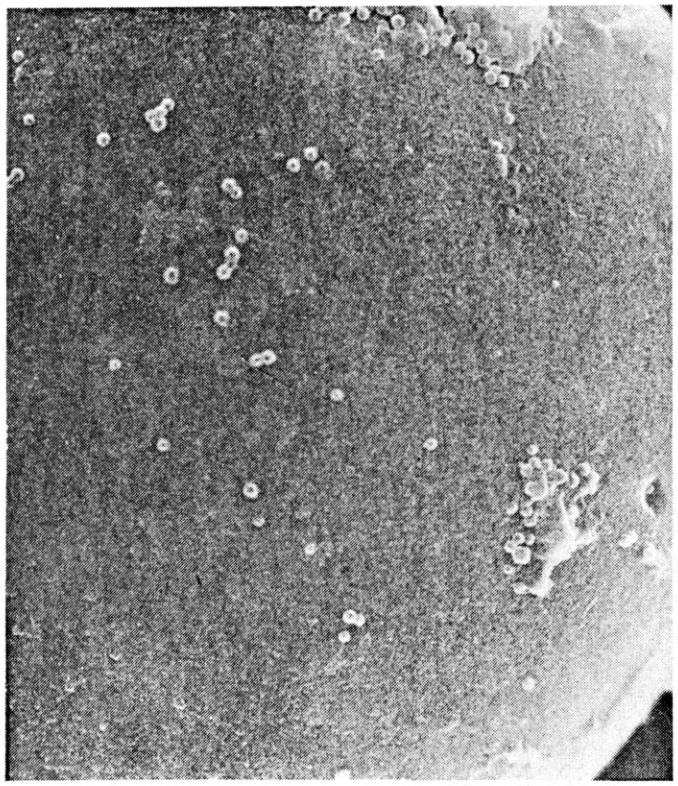
1 mm = 0.60 μm (magnification 2000x)

Figure 10 (a). Scanning Electron Micrograph of the Glass Bead Initially.



1 mm = 0.60 μm (magnification 2000x)

Figure 10 (b). Glass Bead After Attachment of Alum Sol Particles.



1 mm = 0.60 μm (magnification 2000x)

Figure 10 (c). Scanning Electron Micrograph of the Glass Surface After Detachment.

1 mm = 0.60 μm (magnification 2000x)

Figure 10 (d). Scanning Electron Micrograph of Glass Surface After Detachment.

ANALYSIS OF THE RESULTS

All the experiments were performed under identical conditions and the fractional removal versus time was studied for different bed expansions (expanded bed porosity). From Figure 6 we see that maximum fraction removed occurs at 100% expansion. The fluid shear near the particle surface is maximized at this expanded bed porosity ($\bar{\epsilon} = 0.7$) and particle-particle contact are not important because of the buffer effect of surface liquid layers [1,2].

Figure 7 clearly shows that bed expansion of $D_e/D > 2$ does not induce any further removal. The plot of cumulative fraction removed versus expanded bed porosity (Fig. 9) clearly indicates the optimum of 0.68-0.7 (assuming an initial porosity of glass beads = 0.4) which may be excessive in practical design. However, it is of interest to note that the results obtained with this well defined system correlate well with the investigations made by Amirtharajah [1].

A simplistic analysis on time dependence of particle removal was carried out assuming a first order kinetic expression, which can be written as

$$\frac{dN}{dt} = - KN \qquad (14)$$

where 'N' is the number of adhered particles at time 't' and 'K' is the rate constant.

A similar approach has been adopted by Kallay and Matijevic [6], in their studies. Taking x and x_∞ a fractions of particles detached at time t and at infinity, respectively, the integration of Eq. (13) yields

$$\ln (x_\infty - x) = \ln x_\infty - Kt \rightarrow \qquad (15)$$

Figure 8 shows the kinetic plot for the data. The data is comprised of three identical runs at 50% bed expansion. The straight line fits the data very well. The correlation obtained for the three experiments is close to 1 indicating good reproducibility of the experimental runs.

Scanning Electron Micrographs of the glass surface (Fig. 10 (a)) shows a micro rough structure. Figure 10 (b) and 10 (c, d) are pictures of alum sol particles on glass bead before and after detachment, respectively. There is a tendency of particles to flocculate (Fig. 10 (c)) after detachment. Since the alum sol particles are uniform size, it can be inferred that the heterogeneity of the surface will have a significant influence on particle detachment. We also see that complete removal of particles is not possible even under optimum conditions, thus the results agree with the theoretical analysis made by Amirtharajah and Giourgas [3].

CONCLUDING SUMMARY

The study of particle detachment in a fluidized bed was conducted by attaching uniformly dispersed, spherical alum sol particles on glass beads. The results were analyzed using the theories developed by Amirtharajah [1,3]. The results were found to be in good agreement with the theory. It was also determined that even under optimal conditions of backwashing, complete removal by fluidization is not possible. Heterogeneity of the surface influences particle detachment. The model system used confirmed some of the accepted facts in backwashing. The chosen system meets several requirements for a successful analysis and can be used as a useful tool for the study of particle detachment.

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