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RESEARCH PAPER



# Characterization of Filtrate Cakes. Aluminum Hydroxycarbonate

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## ABSTRACT

*Aluminum hydroxycarbonate gel suspensions have been filtered at constant pressure and the experimental data used to analyze and discuss the different procedures for obtaining design parameters such as specific resistance and compressibility of the cake, and resistivity of the filtration medium. It is shown that different algorithms yield different values for these parameters, and the possible pitfalls in the analysis of the data are discussed. The compressibility data of the cake of aluminum hydroxycarbonate have been adjusted to the nonlinear model* $\alpha = \alpha_0(-\Delta P)^s$. *The value of* s *obtained by different mathematical algorithms, 0.34, indicated that the cake is moderately compressible.*

## INTRODUCTION

In the broadest sense, filtration is the separation of suspended particles from a liquid by passage through a porous medium. The separation of a liquid, perhaps containing valuable constituents in solution, from the accompanying solid, which may or may not have value, is an operation which is commonly used in the pharmaceutical and biotechnology industries. The theory of filtration is well established (1,2), but the parameters needed for the scale-up of laboratory filters must be obtained experimentally. It is the purpose of this publi-

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cation to discuss and compare the different methods by which these parameters are obtained, and to indicate the pitfalls to be avoided. The pertinent procedures are illustrated using experimental information on the filtration of aluminum hydroxycarbonate (AHC) obtained by precipitation from aqueous solutions of aluminum chloride (20% weight per volume) with sodium carbonate (40% weight per volume).

Aluminum hydroxycarbonate in gel form is formulated alone or in combination with other actives (like magnesium hydroxide) in a large number of antacid products for the treatment of gastrointestinal problems. Aluminum hydroxide presents antacid activity only when present in an amorphous form, this form being obtained by its precipitation from an aluminum salt with an alkaline carbonate. If the alkaline carbonate is replaced by an alkaline hydroxide the precipitated aluminum hydroxide will be active only when fresh; with time its activity will disappear (3–5). AHC is not absorbed systemically and besides producing constipation does not have undesirable effects on the body. The therapeutic activity of AHC is well documented in the literature (6,7). Chemical and physical characteristics like structure, point of zero charge, and reactivity have been studied in detail (3–5,8–10). Nevertheless, little information is available in the open literature on the physical properties of these gels that will allow a sound design of process equipment, in particular, the pressure filter needed to separate and wash the gels after the precipitation stage.

## THEORY

Filtration is a method of removing suspended solids from a liquid by using a porous medium which retains the particles and passes the clear filtrate. The pressure drop necessary to force the liquid through the cake is usually supplied by use of a pump or by centrifugal force. Filtration is a problem of fluid flow through a porous medium composed of the filter cake and the filter material in series. It is usually accepted that under these conditions the flow is laminar and may be described by Poiseuille's law (1,2):

$$v = \frac{dV}{d\theta} = \frac{A(-\Delta P)g_c}{\mu(R_c + R_m)} \tag{1}$$

That is, the rate of filtration $v$ is proportional to the pressure drop through the combined bed. In Eq. (1) $R_c$ is the resistance of the cake and $R_m$ that of the filter medium. The value of $R_c$ increases during the filtration process and clearly at any given moment will depend on the mass of solids deposited as a result of the passage of the corresponding volume $V$ of the filtrate. If it is assumed that each volume unit of filtrate forms a filter cake of the same volume, we can write:

$$R_c = \frac{\alpha c V}{A} \tag{2}$$

where $c$ is the weight of solids deposited per unit volume of filtrate and $\alpha$ is a proportionality constant known as the specific resistance of the cake. Similarly, if $R_m$ is considered to be equivalent to the resistance of a fictitious layer of filter cake of equal resistance, then:

$$R_m = \frac{\alpha c V_0}{A} \tag{3}$$

where $V_0$ is the volume of filtrate required to form a filter cake of resistance equal to the initial filter medium resistance. Equation (1) may now be written as:

$$\frac{dV}{d\theta} = \frac{(-\Delta P)A^2 g_c}{\alpha\mu c\,(V + V_0)} \tag{4}$$

Equation (4) may be written in the equivalent form:

$$\frac{d\theta}{dV} = \frac{\alpha\mu c}{(-\Delta P)A^2 g_c} V + \frac{\alpha\mu c}{(-\Delta P)A^2 g_c} V_0 \tag{5}$$

Defining two new parameters, $K_p$ and $B$, as:

$$K_p = \frac{\alpha\mu c}{(-\Delta P)A^2 g_c} \tag{6}$$

$$B = \frac{\alpha\mu c V_0}{(-\Delta P)A^2 g_c} \tag{7}$$

we can write Eq. (5) as:

$$\frac{d\theta}{dV} = K_p V + B \tag{8}$$

In certain cases it is necessary to initiate the filtration process with a stage in which the pressure is gradually increased to the desired value (2). If the time required for this stage is $\theta_s$, with the corresponding filtrate volume $V_s$, then Eq. (8) can be integrated to give:

$$\frac{\theta - \theta_s}{V - V_s} = \frac{K_p}{2} V + \frac{K_p}{2} V_0 + B \tag{9}$$

Equation (4) is the basic filtration equation that describes the change of filtrate volume with time, the pressure drop, and the specific resistance $\alpha$. The specific resistance may vary or remain constant during the filtration cycle. If the cake structure and porosity remain constant during the filtration period, the cake is said to be noncompressible. If the cake structure changes because of an increase in density and a decrease in porosity, the cake is said to be compressible. A plot of the filtrate volume against time will provide important information regarding the behavior of the specific resistance with the time of filtration. For example, if filtration is carried on at constant pressure, Eq. (7) indicates that the slope of the curve, $d\theta/dV$, will remain constant for a noncompressible cake and will vary if the cake is compressible. When analyzing this behavior care should be taken of the fact that during the initial moments of filtration $\alpha$ will change, even for a noncompressible cake, because the form of the cake has not been fixed yet.

The following simple equation has been proposed to describe the variation of the specific resistance with pressure drop (1):

$$\alpha = \alpha_0(-\Delta P)^s \tag{10}$$

where $\alpha_0$ and $s$ are adjustable parameters that characterize the cake in question. Equation (10) has a very convenient structure for determining the adjustable parameters $\alpha_0$ and $s$, and can be used over restricted pressure ranges.

In general, the filtration process may be carried out at constant pressure or at constant rate. In the first case the filtrate flow will decrease as the filtration time increases while in the second case the pressure drop will increase as the filtration time increases.

For the case of constant specific resistance, Eq. (8) integrates to:

$$\theta = \frac{K_p}{2} V^2 + BV \tag{11}$$

or

$$\frac{\theta}{V} = \frac{K_p}{2} V + B \tag{12}$$

If the specific resistance varies, then a properly defined average may be used. Equations (11) and (12) allow calculation of the time required to pass a given volume of filtrate.

## Determination of the Filtration Parameters

In general, the easiest way to determine the filtration parameters is to perform the experimental tests at constant pressure drop ($-\Delta P$). In this case the filtrate volume is measured at different times and Eq. (8), or other expressions derived from it, is used to calculate the parameters $K_p$ and $B$. The slope of the function $V(\theta)$, $d\theta/dV$, can be generally determined by adjusting first the function $V(\theta)$ to a second- or third-order polynomial in $V$, without a free term. When doing so, there will be need to eliminate some of the initial points with irregular behavior due to higher flow rates and instability of the incipient cake.

We will now describe some of the possible methods for determining the parameters.

### Optimization

Any of the common optimization algorithms, like simplex and maximum likelihood (11) may be applied to Eq. (8) to determine the best values of $K_p$ and $B$. As usual in this case, there may be several sets of parameters that will give the same degree of adjustment, as judged by the proper statistics (e.g., mean average deviation and root mean square). An alternative procedure may be to fit the data with a second order polynomial on $V$.

### Least Mean Squares (Linear Fit)

Equations (8) and (12) may be fit to a straight line either by graphical means or using a least mean squares algorithm. It should come as no surprise if the values of $K_p$ and $B$ obtained from one of the equations differ from those of the other. The reasons for this anomaly are well described in the literature (12,13). Equation (8) uses the derivative of the original data while Eq. (12) uses a transformed variable. Change of the original variable from $V$ to $d\theta/dV$ or $\theta/V$ will most probably produce a change in the distribution of the error. The only way to establish the best set of parameters is to measure the ability to return the original data. It is a common error to assume that the model is appropriate because the modified data are well described by a straight line.

## Compressibility of the Cake

The values of $K_p$ obtained above, together with Eq. (6), may be now used to determine the specific resistance of the cake from:

$$\alpha = \frac{K_p(-\Delta P)A^2 g_c}{\mu c} \quad (13)$$

The values of the adjustable parameters $\alpha_0$ and $s$ in Eq. (10) may then be determined directly by an optimization algorithm, or by linearization of the equation to:

$$\ln \alpha = \ln \alpha_0 + s(-\Delta P) \quad (14)$$

Again, the parameters obtained should be tested for their ability to recover the original data.

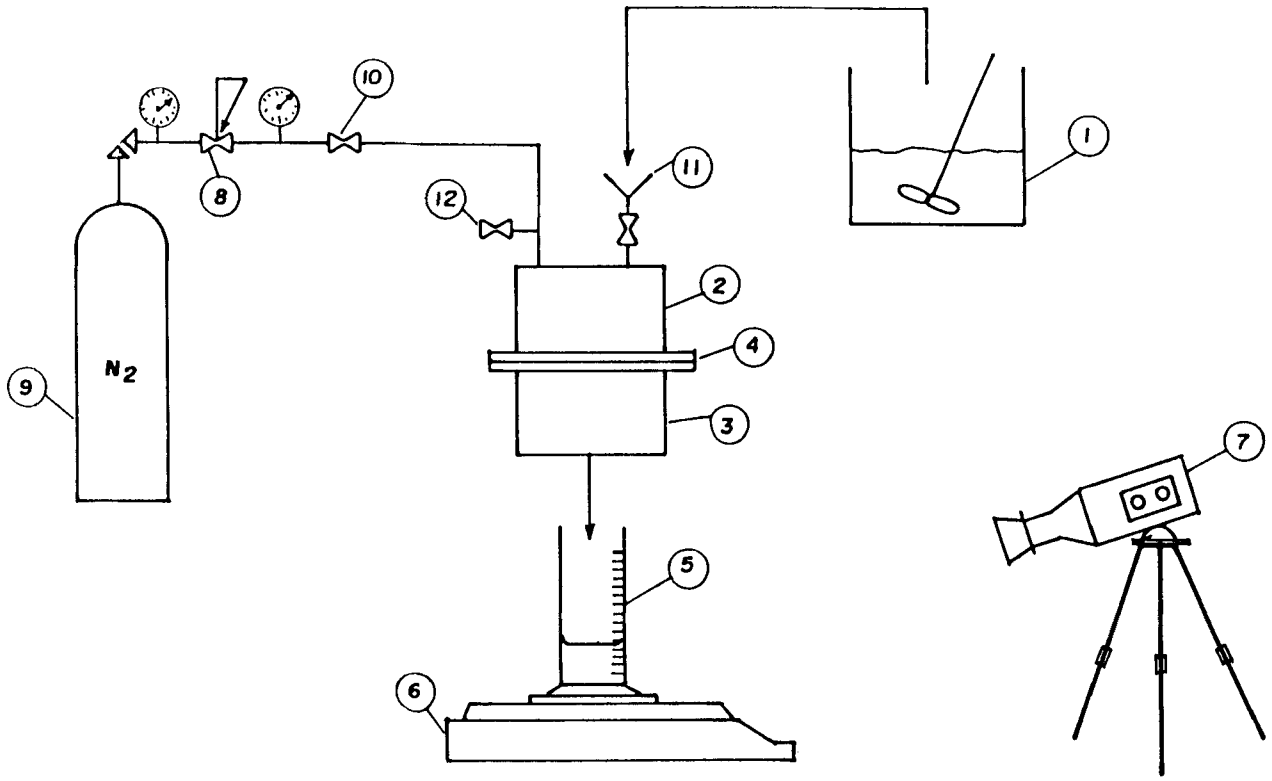
## MATERIALS AND METHODS

Duplicate runs at each pressure (50.66, 131.72, 222.92, and 303.98 kPa) were carried out using a commercial sample of AHC (Química y Farmacia, Ramos Arizpe, México) with an aluminum hydroxide contents of 6.31% (w/v), precipitated from a 20% aluminum chloride solution and a 40% sodium carbonate solution. The AHC suspension contained sodium chloride, as a by-product from the reaction. A polyester cloth (2236-S, LYCOFIL, San Pedro Garza García, México) was used as the filtration medium. Figure 1 is a schematic representation of the filtration system used in the tests. The filtration area was 0.0012469 $m^2$. Filter pressurization was achieved using nitrogen gas from a cylinder equipped with the required pressure regulators and manometers. The filtrate was collected in a 1.0-liter graduated cylinder that was on the weighting dish of a precision balance. Filtration time was taken from the time when the first drop of filtrate appeared until the time when a sudden drop in the working pressure was detected. The wet cake was drained for 3 min, stabilizing the system pressure at 25.33 kPa with nitrogen. Filtration weight and time were monitored with a video camera focused on the graduated cylinder and the digital display of the balance. After a test, replaying of the videotape allowed acquisition of very precise time versus filtrate volume data. The density of the filtrate was determined with the final weights and volumes of the filtrates. With density and weight of the filtrate the accumulated volume was determined at selected times. Room temperature in all runs was 293.15 K. The weight of the AHC cake was determined by drying in a vacuum oven at 318.15 K and 4.15 kPa until constant weight. This quantity was subtracted from the wet weight to obtain the weight of water in the cake. With the filtrate density the concentration of NaCl was determined from tables in the literature (14). This value was used to determine the weight of solution in the wet cake, the AHC dry weight in the cake, and the value of $c$. The filtrate viscosity was obtained from tables for aqueous solutions of NaCl (15).

## RESULTS AND DISCUSSION

Constant-pressure filtration runs were made with duplication at the four pressure drop levels ($-\Delta P$) = 50.66, 131.72, 222.92, and 303.98 kPa. The variation of the filtrate volume with time is given in Table 1 and a typical filtration curve appears in Fig. 2. From this figure it can be seen that the rate of filtration is initially very fast, that it becomes essentially constant after the total filtrate volume has reached about 200 ml, and approaches zero after the total volume has reached about 600 ml. These two features were common to all the pressure levels studied. Figures 3 and 4 illustrate the linearization procedures used to estimate the filtration parameters $\alpha$ and $R_m$, Fig. 3; $\alpha_0$ and $s$, Fig. 4. Additionally, Fig. 3 shows the effect of increasing pressure on filtration rate and also on the values of $\alpha$ and $R_m$, as calculated from the slope and intercept of Eq. (8).

The experimental data given in Table 1, together with the characteristics of the solution and of the suspension, were then used to calculate parameters $K_P$, $B$, and $\alpha$, using Eq. (5) (differential method), Eq. (12) (integral method), Eq. (9) (Svarovsky), and Eq. (11) (maximum likelihood). Similarly, parameters $\alpha_0$ and $s$ in Eq. (10) were calculated by linearization and by the maximum likelihood algorithm. The pertinent results appear in Table 2. Since the range of $s$ is usually between 0.2 and 0.8 (16), the value $s = 0.34$ indicates that the cake is moderately compressible. The statistical data given in Table 2 show that for this particular cake, the data are better fit by the integral method, and that for it, the optimization algorithm gives a smaller relative error. The fact that the differential method gives the largest relative error is not surprising since adjusting the slope of a curve by numerical methods will usually carry a substantial error unless the variable intervals are very small. Further, the optimization algorithm could be used with advantage for the estimation of nonlinear parameters frequently needed in diverse areas of industrial pharmacy, e.g., drug stability testing.




1.- AHC SUSPENSION

2.- TOP OF PRESSURE FILTER

3.- BOTTOM OF PRESSURE FILTER

4.- FILTRATION MEDIUM

5.- GRADUATED CYLINDER

6.- PRECISION BALANCE

7.- VIDEO CAMERA

8.- PRESSURE REGULATOR

9.- NITROGEN CYLINDER

10.- VALVE

11.- ADDITION FUNNEL

12.- VENTING VALVE

**Figure 1.** Experimental setup.

***Table 1***

*Experimental Data, Filtration Time θ (sec), and Filtrate Volume* V *(ml): Duplicate Runs*

| Filtration Pressure (kPa) | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 50.66 | | | | 131.72 | | | | 222.92 | | | | 303.98 | | | |
| $\theta_{1A}$ | $V_{1A}$ | $\theta_{1B}$ | $V_{1B}$ | $\theta_{2A}$ | $V_{2A}$ | $\theta_{2B}$ | $V_{2B}$ | $\theta_{3A}$ | $V_{3A}$ | $\theta_{3B}$ | $V_{3B}$ | $\theta_{4A}$ | $V_{4A}$ | $\theta_{4B}$ | $V_{4B}$ |
| 0 | 0 | 0 | 0.2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3 | 0.5 | 3 | 0.2 | 3 | 2.2 | 3 | 2 | 4 | 19 | 3 | 14 | 3 | 16 | 3 | 19 |
| 4 | 9.4 | 4 | 7.5 | 4 | 27 | 4 | 26 | 5 | 49 | 4 | 41 | 4 | 54 | 4 | 56 |
| 5 | 25 | 5 | 21 | 5 | 49 | 5 | 48 | 6 | 75 | 5 | 72 | 5 | 77 | 5 | 85 |
| 6 | 38 | 6 | 37 | 6 | 71 | 6 | 68 | 9 | 134 | 6 | 91 | 6 | 103 | 8 | 147 |
| 9 | 72 | 8 | 58 | 9 | 118 | 9 | 115 | 12 | 179 | 9 | 145 | 9 | 163 | 11 | 192 |
| 12 | 100 | 9 | 71 | 12 | 156 | 12 | 152 | 15 | 214 | 12 | 186 | 12 | 206 | 14 | 234 |
| 17 | 135 | 12 | 95 | 15 | 184 | 15 | 179 | 18 | 246 | 15 | 222 | 15 | 246 | 17 | 268 |
| 22 | 164 | 15 | 117 | 20 | 228 | 18 | 206 | 23 | 290 | 18 | 257 | 18 | 278 | 20 | 299 |
| 32 | 210 | 20 | 148 | 25 | 264 | 23 | 243 | 28 | 330 | 21 | 280 | 23 | 327 | 25 | 345 |
| 42 | 248 | 25 | 173 | 30 | 297 | 28 | 278 | 38 | 399 | 26 | 320 | 28 | 369 | 30 | 385 |
| 52 | 282 | 35 | 217 | 40 | 355 | 38 | 337 | 48 | 457 | 31 | 358 | 33 | 407 | 35 | 422 |
| 67 | 328 | 45 | 255 | 50 | 404 | 48 | 387 | 58 | 511 | 36 | 393 | 38 | 443 | 45 | 488 |
| 82 | 368 | 55 | 285 | 60 | 448 | 58 | 431 | 68 | 559 | 41 | 424 | 48 | 507 | 55 | 550 |
| 97 | 405 | 65 | 314 | 75 | 508 | 68 | 472 | 80 | 613 | 46 | 453 | 58 | 565 | 67 | 611 |
| 112 | 439 | 75 | 341 | 90 | 563 | 78 | 510 | 110 | 640 | 56 | 506 | 67 | 611 | 97 | 647 |
| 127 | 471 | 90 | 380 | 104 | 610 | 88 | 545 | 140 | 644 | 66 | 556 | 97 | 646 | 127 | 649 |
| 142 | 501 | 105 | 415 | 134 | 634 | 98 | 579 | 170 | 644 | 76 | 602 | 127 | 649 | 157 | 650 |
| 157 | 529 | 120 | 447 | 164 | 637 | 107 | 608 | 200 | 645 | 80 | 620 | 157 | 650 | 187 | 650 |
| 172 | 557 | 135 | 478 | 194 | 639 | 137 | 631 | 230 | 645 | 110 | 647 | 187 | 650 | 217 | 650 |
| 187 | 582 | 150 | 507 | 224 | 639 | 167 | 635 | 260 | 645 | 140 | 649 | 217 | 650 | 247 | 650 |
| 197 | 597 | 165 | 538 | 254 | 640 | 197 | 637 | | | 170 | 650 | 247 | 650 | | |
| 226 | 610 | 180 | 561 | 284 | 640 | 237 | 638 | | | 200 | 650 | | | | |
| 256 | 614 | 198 | 591 | | | 267 | 637 | | | | | | | | |
| 286 | 617 | 228 | 609 | | | 297 | 638 | | | | | | | | |
| 316 | 618 | 258 | 614 | | | | | | | | | | | | |
| 346 | 619 | 288 | 617 | | | | | | | | | | | | |
| 376 | 620 | 318 | 618 | | | | | | | | | | | | |
| | | 348 | 618 | | | | | | | | | | | | |
| | | 378 | 618 | | | | | | | | | | | | |



## CONCLUSIONS

Aluminum hydroxycarbonate gel suspensions have been filtered at constant pressure and the experimental data used to analyze and discuss the different procedures for obtaining design parameters such as specific resistance and compressibility of the cake, and resistivity of the filtration medium. It is shown that different algorithms yield different values for these parameters, and the possible pitfalls in the analysis of the data are discussed. The compressibility data of the cake of aluminum hydroxycarbonate have been adjusted to the non-

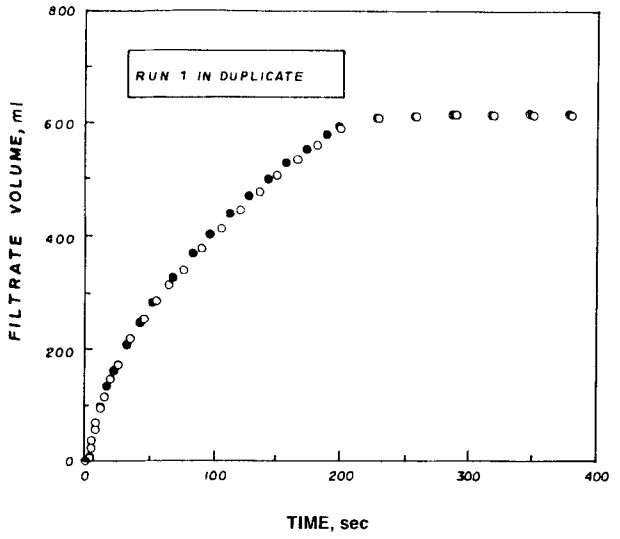



**Figure 2.** Typical filtration run at $(-\Delta P) = 50.66$ kPa, indicating the reproducibility of the data and general behavior.

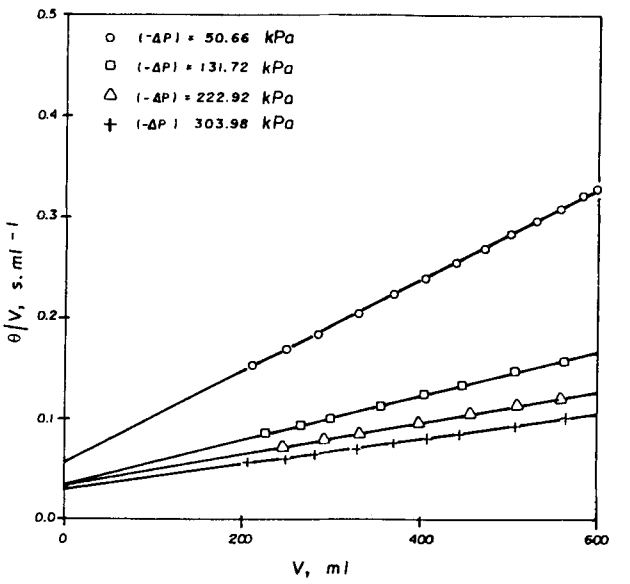



**Figure 3.** Effect of pressure on filtration rate. Linearization method to obtain values of $\alpha$ from the slope, $K_P$ [Eq. (6)] and $R_m$ from the intercept [Eq. (7)].

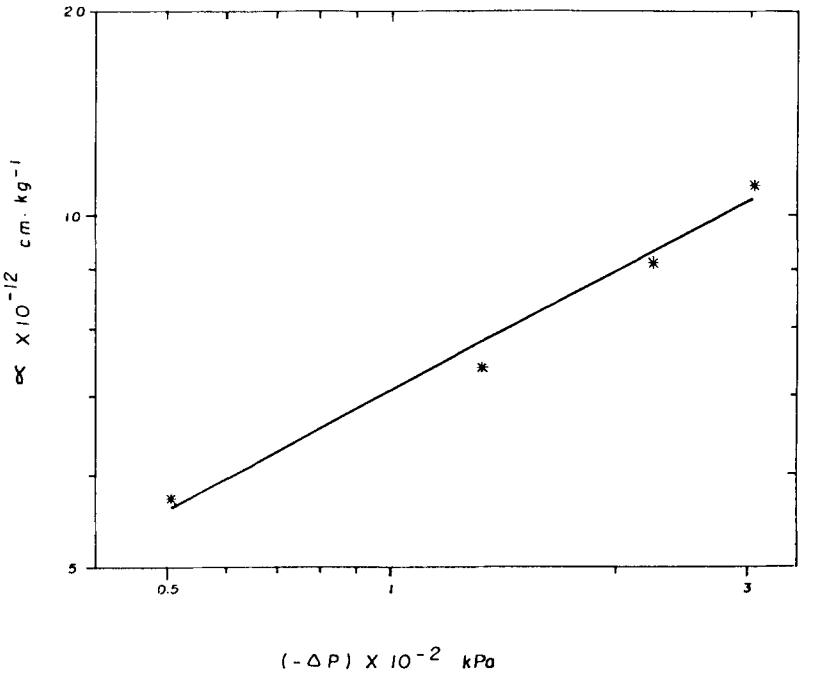



**Figure 4.** Determination of filtration parameters $\alpha_0$ and $s$ by linearization.

*Table 2*

*Characteristic Parameters of the Cake, Calculated by Different Procedures*[a]

| $(-\Delta P)$, kPa | $K_P \cdot 10^4$, $s \cdot cm^{-6}$ | $B \cdot 10^2$, $s \cdot cm^{-3}$ | $R_m \cdot 10^{-8}$, $cm^{-1}$ | $\alpha \cdot 10^{-12}$, $cm \cdot kg^{-1}$ | % Av dev[b] |
|---|---|---|---|---|---|
| | | *A. Differential Method [Eq. (8)]* | | | |
| 50.66 | 9.8 | 3.26 | 1.58 | 6.10 | 5.3 |
| 131.72 | 4.80 | 2.23 | 2.87 | 8.16 | 7.0 |
| 222.92 | 3.54 | 1.58 | 3.42 | 10.38 | 9.5 |
| 303.98 | 3.04 | 1.18 | 3.46 | 12.37 | 8.2 |
| | | *B. Integral Method [Eq. (12)]* | | | |
| 50.66 | 9.19 | 5.81 | 2.81 | 5.73 | 0.34 |
| 131.72 | 4.42 | 3.79 | 4.88 | 7.52 | 0.71 |
| 222.92 | 3.12 | 3.24 | 7.00 | 9.14 | 0.83 |
| 303.98 | 2.62 | 2.81 | 8.22 | 10.66 | 0.83 |
| | | *C. Svarovsky [Eq. (9)]* | | | |
| 50.66 | 4.86 | 3.90 | 1.89 | 6.05 | 1.2 |
| 131.72 | 2.41 | 2.16 | 2.78 | 8.20 | 1.4 |
| 222.92 | 1.90 | 0.69 | 1.47 | 11.11 | 2.5 |
| 303.98 | 1.60 | 0.63 | 1.53 | 12.97 | 1.8 |
| | | *D. Maximum Likelihood [Eq. (12)]* | | | |
| 50.66 | 9.16 | 5.88 | 2.84 | 5.70 | 0.25 |
| 131.72 | 4.30 | 4.03 | 5.19 | 7.31 | 0.67 |
| 222.92 | 3.05 | 3.40 | 7.35 | 8.94 | 0.74 |
| 303.98 | 2.55 | 2.94 | 8.60 | 10.38 | 0.73 |

*E. Specific Resistance [Eq. (10)]*

| Method | $\alpha_0 \cdot 10^{-12}$ $(cm \cdot kg^{-1})$ | $s$ | |
|---|---|---|---|
| Linearization | 7.11 | 0.3393 | 2.5 |
| Maximum likelihood | 7.24 | 0.3403 | 2.1 |

[a]Average of two duplicate runs.
[b]Percent average deviation $= 100 \left| \frac{\text{exptl} - \text{calc}}{\text{exptl}} \right|$.

linear model $\alpha = \alpha_0(-\Delta P)^s$. The value $s = 0.34$ was obtained by different mathematical algorithms, indicating that the cake is moderately compressible.

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