Initial and Final Stages of Compressible Filtercake Compaction

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Filtration experiments were performed to dewater bentonite slurries at different filtration pressures. The diffusivity of the cake was then determined as a function of void ratio using the measured filtration rates, the final void ratios of the cakes, and the theory of compressible filtercakes. Any filtration process can, in theory, be predicted, once the cake diffusivity is known as a function of void ratio and the void ratio as a function of applied pressure. In particular, the final stage of the filtration process can be modeled and offers an alternative route to determine the cake properties from the experimental results. The final slow approach to equilibrium is difficult to study experimentally; other techniques to determine cake properties are therefore preferable. Final stages of the cake compaction are also markedly slower than predictions based on the initial filtration rates. Possible explanations include a filtercake rheology that is more complicated than assumed in the model or time-dependent effects caused by exclusion of ions from the bentonite filtercake, but such explanations appear unsatisfactory. Filtration models in which the filtercake void ratio and permeability depend solely on the imposed stress, may therefore be too simple for some materials.

Introduction

Slurry dewatering is often performed by means of batch filtration at constant pressure. The conventional chemical engineering description of this process distinguishes two stages. During the initial filtration stage, unfiltered slurry remains above the cake. This is followed by expression, during which the filtercake is compacted. These are convenient practical terms, but it should nevertheless be possible to describe the entire dewatering process by a single model if sufficient information is available concerning the properties of the filter cake. Meeten and Sherwood (1994) determined the filtration properties of bentonite slurries from experimental filtration rates measured at the start of filtration. The objective of this article is to compare experiment and theory in both the initial (rapid) and final (slow) stages of dewatering.

Many models of filtration are available, and the work presented here is based on that of Philip and Smiles (1982). The theoretical model is reviewed, and it is shown how information can be obtained from either the initial or final stages of dewatering. Experimental data is considered and the experimental measurements are compared to the predictions of modeling.

Compressible Cake Theory

We consider uniaxial filtration, as depicted in Figure 1. Following Philip and Smiles (1982), we describe both the slurry and filter cake by means of the void ratio e, defined as

$$e = \frac{\text{volume of liquid}}{\text{volume of solid}}. \tag{1}$$

Volume fraction of solids ϕ is related to e via $\phi = 1/(e+1)$.

Filtration occurs sufficiently slowly that the forces acting on the particles and liquid within the cake are always in equilibrium, and we assume that gravity may be neglected. The stress within the cake may be split into a liquid pressure p and the stress within the matrix of solid particles. We further divide the matrix stress into an isotropic part ψ and a deviatoric (shear) stress Σ. The y component of the stress balance within the cake leads to an equation

$$p + \psi + \Sigma_{yy} = \text{constant}. \tag{2}$$

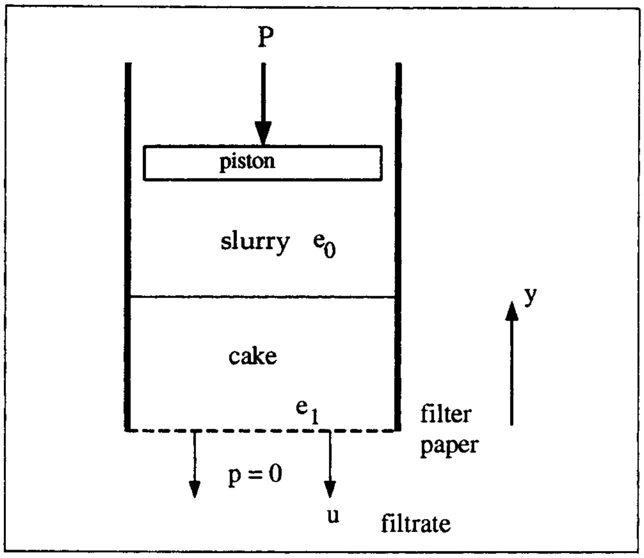

Figure 1. Definition of filtration cell.

During compaction of the cake, the matrix of solid particles is continuously yielding, and Σ_{yy} is related to the yield stress within the cake, as discussed by Meeten (1994). However, the yield stress is generally small compared with the isotropic pressure ψ required to compact the cake, and we choose here to neglect Σ_{yy}. Its inclusion in models of uniaxial compaction is straightforward.

We assume that the void ratio e within the cake is a function of the isotropic matrix stress ψ. Filtrate flow, at a velocity u_{rel} relative to the particles within the cake, is assumed to obey Darcy's law

$$u_{\text{rel}} = -\frac{k(e)}{\mu}\frac{\partial p}{\partial y} \tag{3}$$

where μ is the viscosity of the filtrate, and the permeability k of the cake is assumed to be a function of the void ratio e.

We adopt a Lagrangian coordinate m, defined by

$$m = \int_0^y [1 + e(y)]^{-1} dy. \tag{4}$$

The filtrate is assumed incompressible, and the evolution of void ratio e as a function of time t is given by the equation of continuity

$$\begin{aligned}\frac{\partial e}{\partial t} &= -\frac{\partial u_{\text{rel}}}{\partial m}\\ &= \frac{\partial}{\partial m} D(e) \frac{\partial e}{\partial m}\end{aligned} \tag{5}$$

where

$$D(e) = -\frac{k(e)}{\mu(1+e)}\frac{d\psi}{de}. \tag{6}$$

Equation 5 is a diffusion equation, and $D(e)$ is a diffusivity. In the limit $e \to 0$ (or $P \to \infty$) we expect $k(e) \to 0$ and $d\psi/de \to \infty$: one or the other of these limits will dominate and determine the behavior of $D(e)$. It is most unlikely that $D(e)$ tends to a finite, nonzero value as $e \to 0$.

At time $t = 0$, the pressure P is applied to the upper surface of the slurry, and hence, within the cake, Eq. 2 becomes

$$p + \psi(e) = P. \tag{7}$$

Initially, the slurry has void ratio e_0, and this will usually be sufficiently large (that is, the solids volume fraction ϕ is sufficiently small) that $\psi(e) \simeq 0$, and hence the liquid pressure $p \simeq P$ far from the filter cake. We shall assume that P is held constant throughout the filtration process.

At the base of the cake, the filtration is at atmospheric gauge pressure $p = 0$, and hence the matrix stress $\psi = P$. The immediate effect of the applied pressure is therefore to compact the slurry at the base of the cake to a void ratio e_1, where

$$\psi(e_1) = P. \tag{8}$$

If the volume of slurry is infinite, there are solutions of Eq. 5 in which the cake thickness grows as $t^{1/2}$ and the filtration flux decreases as $t^{-1/2}$. Such solutions also hold during the early stages of dewatering of a finite sample of slurry, when the upper piston (see Figure 1) is sufficiently far from the cake. The void ratio e can be expressed as a function of a similarity variable $s = mt^{-1/2}$, and the diffusion Eq. 5 becomes

$$\frac{d}{ds}\left(D(e)\frac{de}{ds}\right) = -\frac{s}{2}\frac{de}{ds}. \tag{9}$$

The volume of filtrate Q collected per unit area of filter paper is given by

$$Q = St^{1/2} \tag{10}$$

where

$$S = \int_0^\infty (e_0 - e) ds \tag{11}$$

is a desorptivity.

A straightforward modification of the results of van Duyn and Peletier (1977) leads to the bounds

$$2\int_{e_1}^{e_0} (e_0 - e) D(e) de \le S^2 \le 2(e_0 - e_1) \int_{e_1}^{e_0} D(e) de. \tag{12}$$

An inequality such as Eq. 12 cannot be differentiated, since S^2 might oscillate rapidly between the upper and lower bounds, and hence the derivative of S^2 might bear no relation to the derivatives of the bounds. In practice, however, we expect that S and $D(e)$ are well behaved, and the bounds are tight. Differentiation of Eq. 12 leads to the approximate expression for the diffusivity given by Smiles and Harvey (1973)

$$D(e_1) = \frac{-1}{2(e_0 - e_1)}\frac{dS^2}{de_1} - \frac{rS^2}{2(e_0 - e_1)^2}. \tag{13}$$

The parameter r in the final term of Eq. 13 lies in the range $0<r<1$, and the choice $r=2-\pi/2=0.429$ makes Eq. 13 exact in the case when D is constant. However, this final term is typically small when the filtercake is compressible. Analysis of the experiments reported below was performed with $r=0.429$.

If the volume of slurry is finite, with $0 \le m \le M$, the boundary condition $u_{\text{rel}}=0$ is appropriate at the upper surface of the slurry, and hence

$$\frac{\partial p}{\partial m}=\frac{\partial \psi}{\partial m}=0 \quad \text{at} \quad m=M. \tag{14}$$

During the early stages of filtration, when the piston is far from the surface of the cake, the similarity solution (Eq. 9) holds. Eventually, however, the supply of slurry above the cake becomes exhausted, and filtration ceases when all the slurry has been compacted to a void ratio e_1. In the final stages of filtration, at time $t \ge t_f$ when the cake is almost uniform, the diffusivity may, to a first approximation, be considered uniform throughout the cake with $D(e) \simeq D_1 = D(e_1)$. The diffusion Eq. 5, together with boundary conditions (Eqs. 8 and 14), then has the solution (Carslaw and Jaeger, 1959)

$$e(m)=e_1+\sum_{n=0}^{\infty} A_n \sin\left[(2n+1)\frac{\pi m}{2M}\right] \times \exp\left[-\frac{(2n+1)^2\pi^2}{4M^2}D_1 t\right], \tag{15}$$

where the coefficients A_n may be obtained from a Fourier series expansion of the void ratio distribution at time t_f. The rate of fluid loss (per unit area) through the filter paper is

$$\begin{aligned}\frac{dQ}{dt}&=-u_{\text{rel}}(0)\\ &=D_1\left.\frac{\partial e}{\partial m}\right|_{m=0}\\ &=\sum_{n=0}^{\infty} D_1 A_n (2n+1)\frac{\pi}{2M}\exp\left[-\frac{(2n+1)^2\pi^2}{4M^2}D_1 t\right].\end{aligned} \tag{16}$$

At large times, the series (Eq. 16) converges rapidly, and terms $n>0$ may be neglected. The rate of fluid loss takes the form

$$\log\left(\frac{dQ}{dt}\right)=C-\frac{\pi^2 D_1}{4M^2}t \tag{17}$$

for some constant C. If $Q_{\max}$ is the total amount of fluid squeezed from the cake before filtration ceases, Eq. 17 may be integrated to give

$$Q_{\max}-Q=\frac{4M^2\exp(C)}{\pi^2 D_1}\exp\left(-\frac{\pi^2 D_1}{4M^2}t\right). \tag{18}$$

The mass of filtrate collected is $W=\rho Q$, where ρ is the filtrate density, and hence

$$\log(W_{\max}-W)=C+\log\left(\frac{4M^2\rho}{\pi^2 D_1}\right)-\frac{\pi^2 D_1 t}{4M^2}, \tag{19}$$

where $W_{\max}=\rho Q_{\max}$. We shall see later (Figure 5) that numerical solutions of the nonlinear Eq. 5 do indeed exhibit the asymptotes (Eqs. 18 and 19) in the final stages of compaction.

Experimental Measurement of Cake Properties

Experiments were performed in a standard API filter-cell (American Petroleum Institute, 1988) at various pressures P, using an aqueous suspension of Bentopharm (a pharmaceutical grade bentonite, Bromhead and Denison Ltd., UK), and an initial void ratio $e_0=43.5$. The cake void ratio e_1 at the end of each test, when filtration had ceased, was determined by weighing the cake, first while wet, and subsequently after drying for 24 h at 105°C. In the subsequent computation of void ratio of the suspension and of the cakes, the density of the dry clay was taken to be 2,600 $\text{kg}\cdot\text{m}^{-3}$. The resulting values for e_1, shown in Figure 2, were fitted to the expression

$$e_1=e_\infty+\left(\frac{p_1}{P+p_2}\right)^{\alpha} \tag{20}$$

with $e_\infty=0.45$, $p_1=18.6$ bar, $p_2=0.15$ bar, and $\alpha=0.60$. Equation 20 with $p_2=0$ is of the form found by Carrier and Beckman (1984) and by Den Haan (1992). The addition of a fourth parameter p_2 markedly improves the fit to the experimental data shown in Figure 2, but it should not be assumed that Eq. 20 has any fundamental significance, nor that it may be extrapolated beyond the range of pressures over which experiments were performed. In particular, Eq. 20 predicts $e_1 \to e_\infty+(p_1/p_2)^{\alpha}=18.5$ as $P \to 0$, rather than $e_1 \simeq e_0 = 43.5$.

In each experiment, the filter cell of area 4,560 mm^2 was loaded with 50 g slurry and the mass of filtrate was measured as a function of time. Results are shown in Figure 3, and were converted into volumes assuming a filtrate density $\rho=1{,}000\ \text{kg}\cdot\text{m}^{-3}$. The filter paper was supported by a wire mesh, below which was a dead volume which had first to be filled before filtrate emerged from the apparatus. The results of Figure 3 were therefore corrected to ensure that all curves

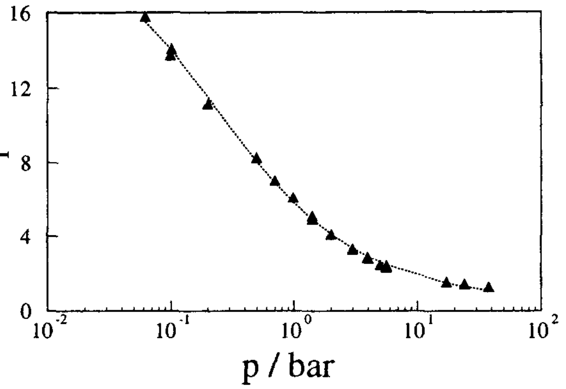

Figure 2. Experimentally measured final void ratio e_1 plotted against the filtration pressure P.

Dotted line shows the fitted curve (Eq. 20).

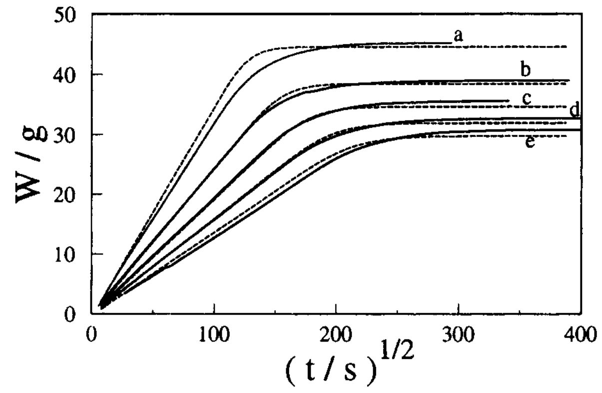

Figure 3. Mass W of filtrate collected from API cell at pressures: (a) 5.7 bar; (b) 0.5 bar; (c) 0.2 bar; (d) 0.1 bar; (e) 0.05 bar.

—— experimental results; ---- predictions obtained by integrating Eq. 5 numerically.

pass through the origin. The wire mesh was slightly flexible, and the correction varied from 1.2 g (at filtration pressure $P = 5.7$ bar) to 3.4 g (at $P = 0.05$ bar). From the early straight-line portion of each curve, we may determine the desorptivity S, and values for $S(e_1)$ are shown in Figure 4. The diffusivity $D(e)$ may then be estimated by means of Eq. 13, from which we see that S must tend to zero sufficiently rapidly as $e_1 \to e_0$ if $D(e)$ is to be well-behaved as $e \to e_0$. The initial void ratio of the slurry was $e_0 = 43.5$, and a least-squares fit to the data in Figure 4 was obtained by the expression

$$S(e_1) = A(e_0 - e_1)(B - e_1)^{\beta} \tag{21}$$

with $A = 16.6\ \mathrm{pm \cdot s^{-1/2}}$, $B = 78.8$, and $\beta = 2.68$. Once again, there is no fundamental reason for choosing the expression (Eq. 21): it merely represents a convenient fit to the data, and is valid only for the initial void ratio $e_0 = 43.5$ used in the experiments. Inserting Eq. 21 in Eq. 13, we obtain the approximation

$$D(e) = C(E - e)(B - e)^{2\beta - 1} \tag{22}$$

where $C = 955\ \mathrm{pm^2 \cdot s^{-1}}$, and $E = 51.5$. Note that Eq. 22 predicts that D tends to a finite, nonzero value $CEB^{2\beta-1}$ as $e \to 0$, which is not expected from Eq. 6. This serves as a warning that an expression such as Eq. 22 should not be extrapolated beyond the range over which the experimental data were obtained.

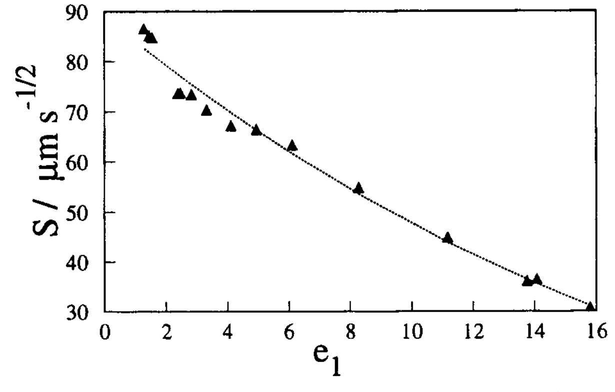

Figure 4. Experimentally measured desorptivity S plotted against the final void ratio e_1.

Dotted line shows the fitted curve (Eq. 21).

The nonlinear diffusion Eq. 5 may now be solved numerically, using Eq. 22 for the diffusivity $D(e)$ and Eq. 20 for the boundary condition (Eq. 8) at the filter paper. The predicted fluid loss W is shown in Figure 3. The difference between the experimental and numerical results for the final amount of filtrate is at most 1.2 g ($P = 0.05$ bar), which is less than 3% of the total amount collected. However, close inspection suggests that the rate at which the computed results achieve their final steady state is too rapid. We can examine this in more detail. Figure 5 shows numerical predictions of the mass of fluid yet to be expressed, as a function of time. Also indicated on Figure 5 are the asymptotic slopes given by Eq. 19, which are in good agreement with the full numerical results when t is large.

We now study the experimental results, in order to determine the actual rate of approach to the final steady state. The mass of fluid yet to be expressed may be estimated by subtracting the mass of filtrate $W(t)$ collected at time t from the maximum amount collected $W_{\max}$. In practice, the subtraction of two nearly equal numbers leads to low accuracy, and results were affected by slow processes such as evaporation of the collected filtrate, which caused a weight loss of approximately 1.4 $\mu\mathrm{g \cdot s^{-1}}$. The results shown in Figure 6 include corrections for evaporation. The addition of individual drops to the collected filtrate can clearly be seen.

Equation 19 predicts that $\log(W_{\max} - W)$ should be linear in t for sufficiently large t, and this was observed in the numerical computations shown in Figure 5. The experimental results shown in Figure 6 exhibit such behavior for 10 g > $(W_{\max} - W) >$ 1 g. Deviations for large t (such as curves d, e) are presumable due to the incorrect choice of $W_{\max}$ when analyzing the data. The major difference between the experimental results of Figure 6 and the predictions of Figure 5 is the rate at which the final steady state is achieved. The two figures are drawn to the same scale, and the slopes of the experimental curves are approximately half those of the nu-

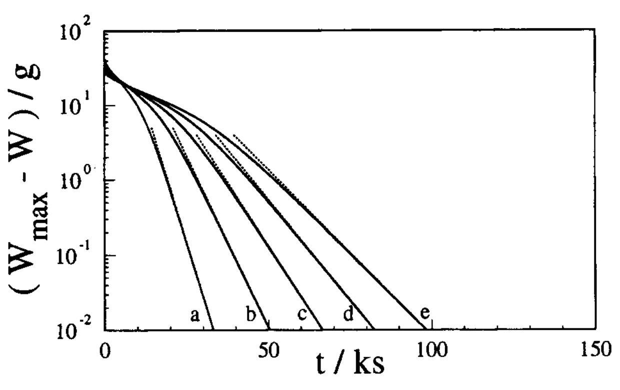

Figure 5. Numerical results of Figure 3 replotted to show the mass of filtrate $W_{\max} - W$ to be collected.

Filtration pressures as in Figure 3. —— full numerical solution of Eq. 5; ····· asymptotes predicted by Eq. 19.

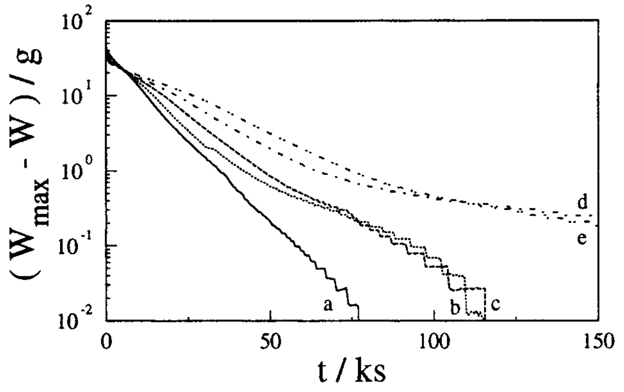

Figure 6. Mass of filtrate $W_{max} - W$ to be collected from final stages of filtration experiments of Figure 3 at pressures; (a) 5.7 bar; (b) 0.5 bar; (c) 0.2 bar; (d) 0.1 bar; (e) 0.05 bar.

merical predictions. This difference in the rate of approach to the final steady state can also be seen in Figure 3.

Discussion

The rate of loss of filtrate from a compressible filtercake is largely determined by the region of low permeability at the base of the cake. In the analysis presented above, it is assumed that the void ratio at the base of the cake jumps instantaneously to the value (Eq. 8) at the start of filtration, and subsequently does not vary. However, there is evidence (Meeten and Sherwood, 1994) that void ratios measured after filtration has ceased (as here) differ from void ratios measured at the base of dissected, nonuniform cakes which are still growing (Meeten, 1993). This can also be seen in concentration profiles determined in cakes allowed to grow for different times before dissection (Sherwood et al., 1991a).

One explanation might be the ability of a clay compact to act as an ion selective membrane (Kharaka and Berry, 1973). During early stages of filtration, the filtrate will be relatively free of salt; towards the end of filtration, the salinity will be higher (Bolt, 1961a,b). The final, higher salinity will compress electrical double layers, leading to a cake with a lower void ratio and permeability. However, estimates of the decrease in void ratio at the base of the cake due to this mechanism are typically only 10% (Sherwood, 1992) and the estimated diffusivity (Eq. 22) does not suggest that the decrease in e will cause a decrease in $D(e)$.

Another explanation might be long-term creep of the cake. Soils containing clay are known to exhibit creep, although the strain rates quoted by Mitchell (1976) are typically very low. Once again, this mechanism will tend to decrease the void ratio, and we have no evidence that this will lead to a decrease in $D(e)$.

The analysis assumes that the rate of compaction of the cake is controlled by the rate at which fluid is expressed, rather than by any resistance to deformation of the matrix of solid particles. Stress relaxation at the end of squeeze-film experiments on filter cakes was investigated by Sherwood et al. (1991b). Rapid relaxation over times of the order of 100 s was observed when $e \simeq 1$. However, slower relaxation mechanisms were also present, and a stretched exponential gave the best fit to results obtained over 300 s. One method to test the relative importance of fluid and matrix response to the applied stress might be to change the fluid viscosity. However, the properties of clay slurries are strongly influenced by clay-water interactions, and these would be modified by the presence of any added viscosifiers. Alternatively, the initial quantity of clay suspension placed within the cell might be changed: the asymptote (Eq. 19) should still hold if the expression of filtrate controls the relaxation towards a uniform cake. Buscall and White (1987) and Sherwood et al. (1991a) suggest possible rheological models for the matrix. Although forward modeling of the compaction of filter cakes would be straightforward for many rheological models of the matrix, little is known of how the techniques used above to determine cake filtration properties from experimental data may be extended to include systems in which the matrix rheology is important.

A much simpler explanation might be that relaxation mechanisms, or fines migration, reduce the cake permeability over long timescales but have little effect upon the cake compressibility. The cake diffusivity $D(e)$ would be smaller at the end of the test than at the beginning.

Conclusions

The filtercake diffusivity $D(e)$ can in principle be determined from the final stages of filtration experiments. However, the experimental difficulties are such that the method is not to be preferred over other techniques. Results for $D(e)$ obtained during the final stages of filtration were typically only half those obtained from the initial stages, and modeling based on the latter values therefore predicts that the final expression of filtrate is faster than observed in the experiments. The origin of this is unclear, and might simply be due to fines migration or particle rearrangement which reduces the cake permeability in the final stages. Alternatively, creep of the matrix of clay particles may play a role in the final stages of compaction. Our understanding of filtercake rheology is not yet sufficiently complete to allow us to distinguish slow rheological processes within the matrix of particles, from slow deformations due to the flow of filtrate through the low permeability filtercake.

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Manuscript received July 29, 1996, and revision received Jan. 17, 1997.