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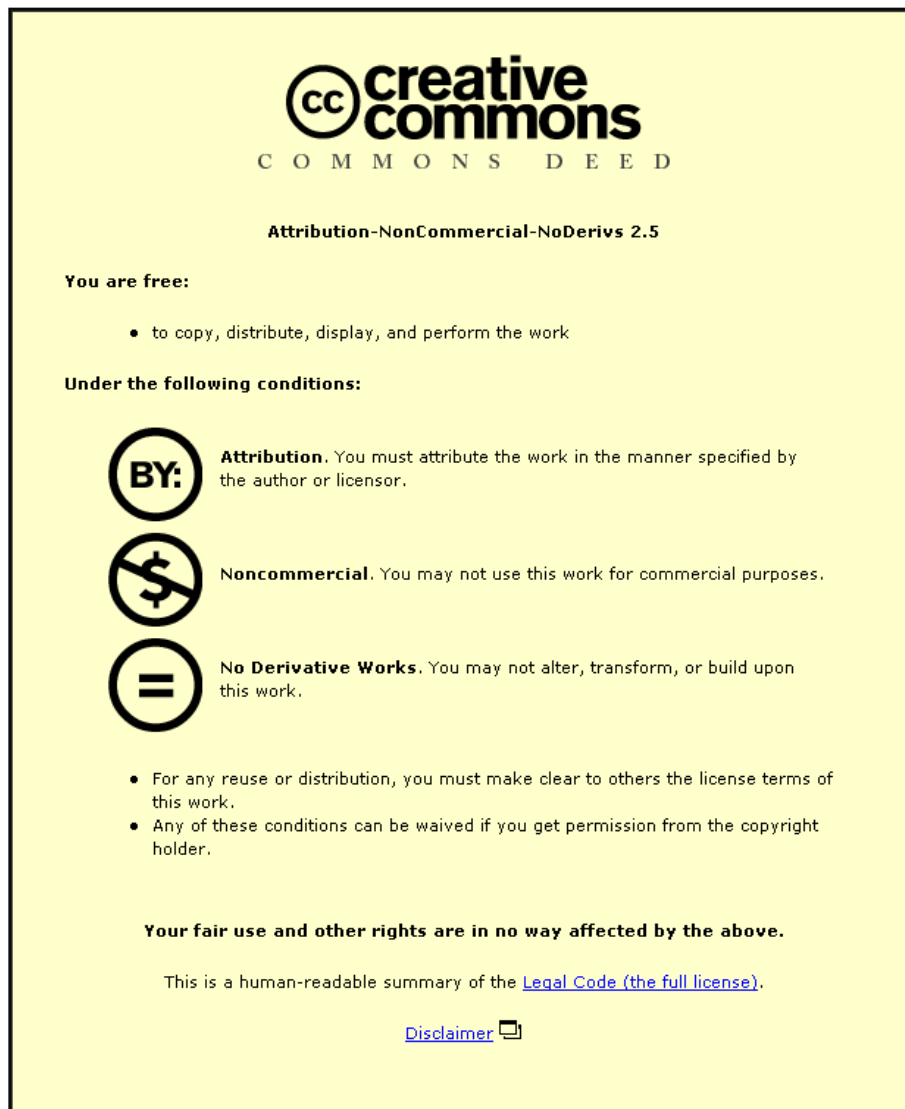
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THE ROLES OF PARTICLE AND LIQUID PROPERTIES IN EXPRESSION - AN EXPERIMENTAL STUDY

R.J. Wakeman and E.S. Tarleton (e.s.tarleton@lboro.ac.uk)

Separation Processes Centre, University of Exeter, North Park Road, Exeter, Devon, EX4 4QF, UK.

ABSTRACT

The objectives of this work were to study effects of particle characteristics on the kinetics of the compression, consolidation and drainage processes. Work has been carried out on a range of solid/liquid mixtures; in each case the mixture was characterised in respect of its particle size and shape, the zeta potential associated with the particle and the ionic strength of its solution environment, and the resulting aggregation of the particulate phase. Expression experiments were carried out in a specially designed piston press and in a scaled-down version of an industrial horizontal tube press. Subsidiary experiments included numerous jar sedimentation tests and a limited number of filtration tests. The data presented highlight the important experimental parameters and serves to illustrate those factors which should be considered by the process engineer when selecting/designing separation equipment.

INTRODUCTION

Variable volume filters are often used commercially to separate fine particle/liquid systems. The most important industrial groups being the tube press and the membrane plate filter where the filtration chamber is divided into two compartments by an impermeable elastic membrane. Pressure applied in one compartment causes the membrane to expand and squeeze the other chamber previously filled with a batch volume of suspension. When the resultant cake formation and compression at the filtering surface are complete the pressure is released, cake discharged and the cycle repeated. Although filters of this type can produce high output rates per unit filter area at close to optimum cake thickness, problems can occur if the solution environment of the process suspension is not carefully controlled. For example, on occasions the wetness of filter cakes can be inexplicably high or unreasonably high filtration pressures and long times are required to produce the desired product. Thus, there is a need to recognise and understand those factors which can influence the filtration, compression and consolidation mechanisms and identify how these might be favourably controlled. To meet these goals it was necessary to design and construct equipment capable of following expression as a dynamic process to obtain information on cake formation, particle packing and drainage rates. This paper considers some of the implications of the work performed and emphasises the information of relevance to the separations technologist.

PISTON PRESS TESTS

Numerous suspension/expression tests have been carried out using the piston press shown schematically in Figure 1. The equipment comprised a stainless steel expression cell of internal diameter 43 mm and height 198 mm fitted with a suitable membrane. The cell was filled at the start of the test with a prepared suspension and constant pressure applied via a pneumatically operated piston arrangement allowed expression to proceed. Both the volume of permeate and piston displacement were monitored continuously throughout an experiment to allow the transient effects to be recorded. The equipment was used to investigate the influence of the following parameters:

- a) Initial concentration of the suspension

- b) Expression pressure
- c) Particle size
- d) Electrophoretic mobility of the particles
- e) Ionic strength of the solution environment.

The electrophoretic mobility (EPM) was measured as a function of the pH for each suspension used; the EPM is directly proportional to the zeta-potential and hence varies with the magnitude of the electrical charge surrounding the particle. The EPM-pH relationship is measured and used in this work as an interpretative tool, as it is otherwise considerably more difficult to show quantitatively the effects of (d) and (e) above. The effects of (e), combined with chemical adsorption equilibrium at the particle-solution interface, leads to the development of a surface electric charge which manifests itself as a force (usually repulsive) between the particles. The EPM is a measure of the repulsive force. In this work attempts have been made to use existing equations developed from models in the colloid science literature to quantify the attractive and repulsive interactions between particles, but results from these models do not appear reliable because of assumptions made in their initial development. For these reasons, and because it is really the influence of the interactive forces that are being demonstrated by this work, parameters (d) and (e) above are combined and shown as a pH effect in what follows.

The materials used in this work are shown in Table 1.

Aqueous suspensions were used throughout the work, each suspension being made from double distilled water and the pH then being adjusted to that required for the particular experiment with indifferent electrolyte. In some tests electrolyte (NaCl or CaCl₂) solutions were used.

Effects of Particle Size

The effect of particle size was to influence the rate of expression; coarser particle suspensions are expressed more readily than finer ones, and are subject to settling during expression. For example, 15 μm calcite particles in a 0.09 volume fraction suspension were fully expressed within 20 s whilst a 3 μm suspension required 2000 s under the same conditions. As the particle size is decreased interparticle repulsive forces become appreciable compared with gravitational and imposed mechanical forces, and the porosity of the deposit is higher. The final moisture content of the compact is affected to only a small extent by particle size provided the imposed force is sufficiently large to overcome the repulsive force.

Effects of Feed Concentration

The effect of suspension concentration was to modify the rate of expression, with lower rates being found at higher concentrations (Figures 2 and 3). This is largely due to the more rapid formation of a thicker cake, and increased surface interaction thereby increasing the resistance to fluid flow sooner in the expression process. A significant number of experiments suggested that there was an effect of initial concentration on the final solids volume fraction in the cake (e.g. Figure 2); this has been observed in pressure filtration previously and explained by the different modes (bridging and blocking) of particle deposition at the filter medium. There is no direct evidence for such explanations, and they do not account for the result shown here where a minimum volume fraction is found in the cake at an intermediate feed concentration.

Effects of Pressure

Expression pressure (varied up to ~45 bar in this work) has a major effect on some suspensions, but a fairly minor effect on others. Higher pressure generally leads to the more rapid formation of a drier cake, but above a certain pressure only marginal improvements in rate are obtained and no further reduction in cake moisture contents are found. Two examples of the effects of pressure are shown in Figures 4 and 5; Figure 4 indicates only marginal differences in the rate of dewatering

magnesium carbonate suspensions, but after 1500 s the approach to the final cake composition is very slow (a constant composition being reached after several days) and the moisture content is strongly dependent on the applied pressure. (A similar effect of pH is shown on Figure 8). Figure 5 shows a different situation where pressure affects the rate of expression to a greater extent than it does the final moisture content. The reasons for these different behaviours lie with the range over which the interparticle repulsive forces extend, hydromagnesite is a composite of MgCO_3 , $\text{Mg}(\text{OH})_2$ and water of hydration, and the magnitude of the pressures existing between the particles at high separation distances in suspension (400 mm at 90.4 volume fraction) presents a problem as it might only be explained by (a) a double layer charge being given by the lattice charge of the mineral and (b) a thick layer of structured water at the particle surface. The diffuse double layer then originates at this distance from the surface. The lattice charge would need to correspond to a much higher surface potential than zeta potential measurements would suggest. It is difficult to reconcile the magnitude of the interparticle pressures observed with expectations from double layer theory when applied to the hydromagnesite data.

Effects of pH

Effects of pH on expression are shown on Figures 6 to 9. The trends shown on each figure are similar, although the shape of the curves tends to vary. The magnesium carbonate data are discussed above. Calcite has a low zeta potential ($< |20|$ mV) and the repulsive forces are correspondingly low hence the rapid expression shown on Figure 6 and the negligible effect of pH on either the rate of the final cake moisture content. Figures 7 and 9 both show that more rapid expression occurs as the pH approaches the point of zero charge (pzc) where interparticle repulsions do not exist. The data in Figure 10 are more extensive and shows that the fastest expression rate occurs at the pzc (pH 4.12); at higher pH's the double layers are thick and repulsive forces are high; at lower pH's the ionic strength is high, causing compression of the double layer and the existence of repulsive forces of a slightly lesser magnitude. The effect of pH on the final moisture content is a secondary one provided the applied force is sufficiently greater than the repulsive forces.

TUBE PRESS TESTS

The above observations have been confirmed by experiments using a scaled-down horizontal tube press (as supplied by VC Filters, Huddersfield). The tube press filtration surface was 235 mm long by 128 mm diameter, and the thickness of the suspension holding volume was 13.5 mm. The main difference between this and the piston press used above (apart from cake formation on a cylindrical rather than a planer surface) is the small depth of suspension at the start of a test, 13.5 compared with 198 mm. Final cake moisture contents from the tube and piston presses were similar for tests at similar conditions.

CONCLUSIONS

There are several conclusions to be drawn from existing colloid science literature, and which have been supported by this work:

1. Particle-particle interactions increase with the solids volume fraction.
2. The ionic strength of the solution plays an important role (through the double layer thickness) in determining the magnitude of the interactions.
3. Reducing the magnitude of the repulsive force causes the dispersion to become unstable, and generally more easily separated.

4. Repulsive forces can be reduced by either (a) adding a non-adsorbing electrolyte to the solution to change the distribution of solution ions around the particle, or (b) altering the electrical charge on the surface of the particle by the specific adsorption of certain ions.

The following conclusions which may be drawn from this experimental programme carry additional implications for the process engineer:

5. The iso-electric point of metal oxides is very sensitive to small amounts of adsorbed impurities.
6. Around the iso-electric point the following should be expected

- a) faster settling rates,
- b) most rapid filtration/expression, and
- c) higher moisture content cakes/sediments, *

due to aggregation of particles when the repulsive forces are very small.

7. At maximum or minimum zeta potentials the following should be expected:

- a) slower settling rates,
- b) slower filtration/expression rates, and
- c) lower moisture content cakes/sediments, *

due to the existence of greater repulsive forces maintaining the particles better dispersed through the suspension.

8. An important relationship which is likely to be of great assistance in the evaluation of particular separation problems is the EPM (or zeta-potential) vs. pH plot. This, together with particle size, should be measured as a matter of course.
9. The compression of thinner suspension layers enables lower moisture content cakes to be obtained more readily; conclusions 5 to 7 are more pertinent to variable chamber filter presses and to some designs of belt press filters than to tube presses, because of the thicker suspension layers which are often employed in those machines.

* Effects of zeta-potential on moisture contents are secondary when sufficiently higher applied pressures are used - effects of zeta-potential on rates are primary at all applied pressures.

ACKNOWLEDGEMENT

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FIGURES AND TABLES

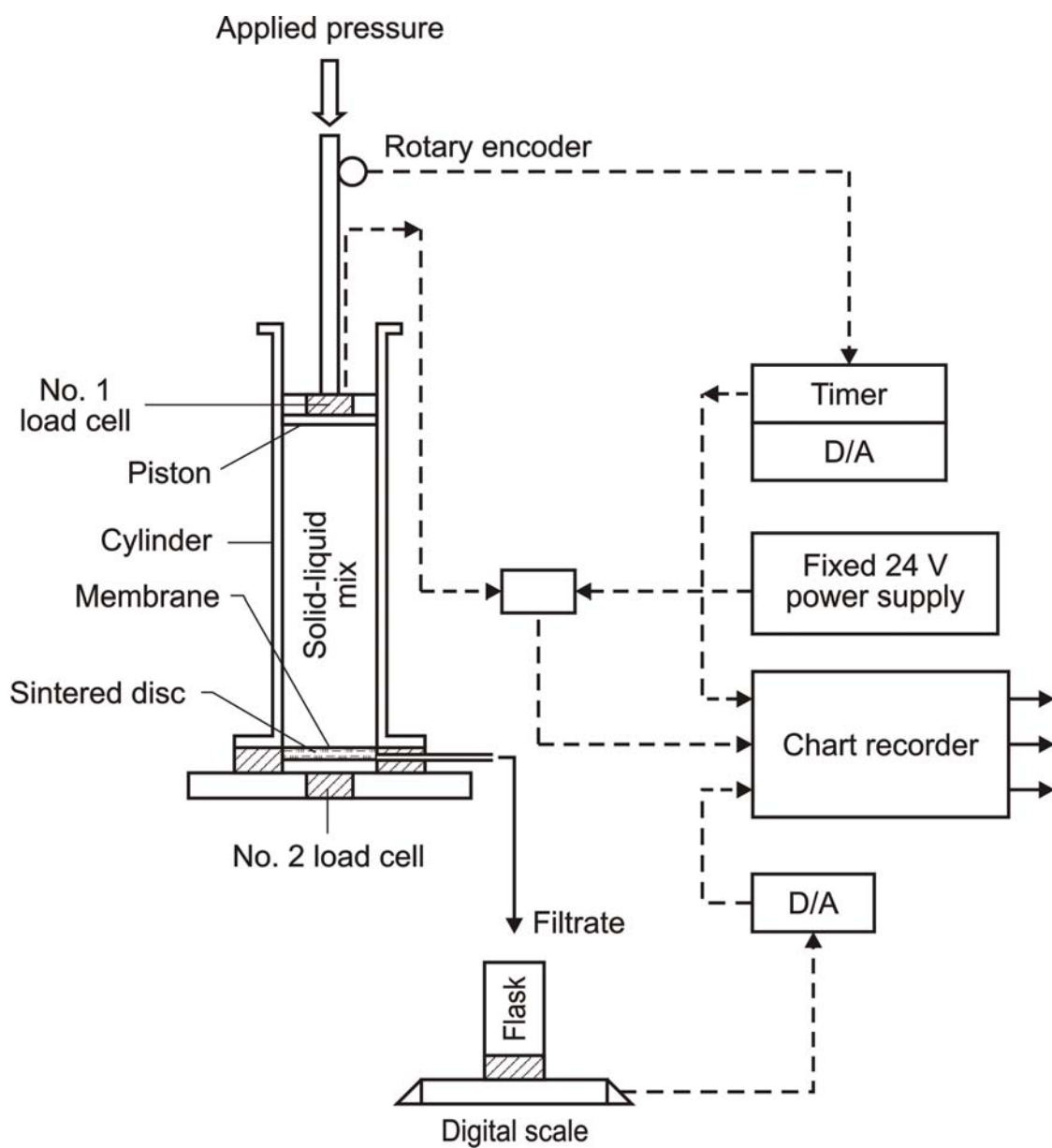


Figure 1: The experimental equipment.

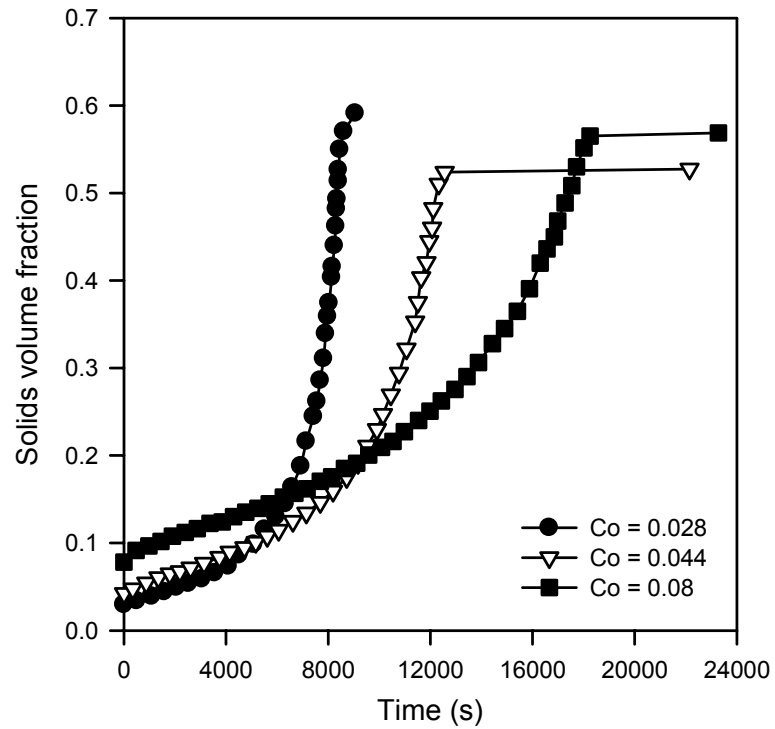


Figure 2: Expression of anatase at pH 7.5 and 42 bar.

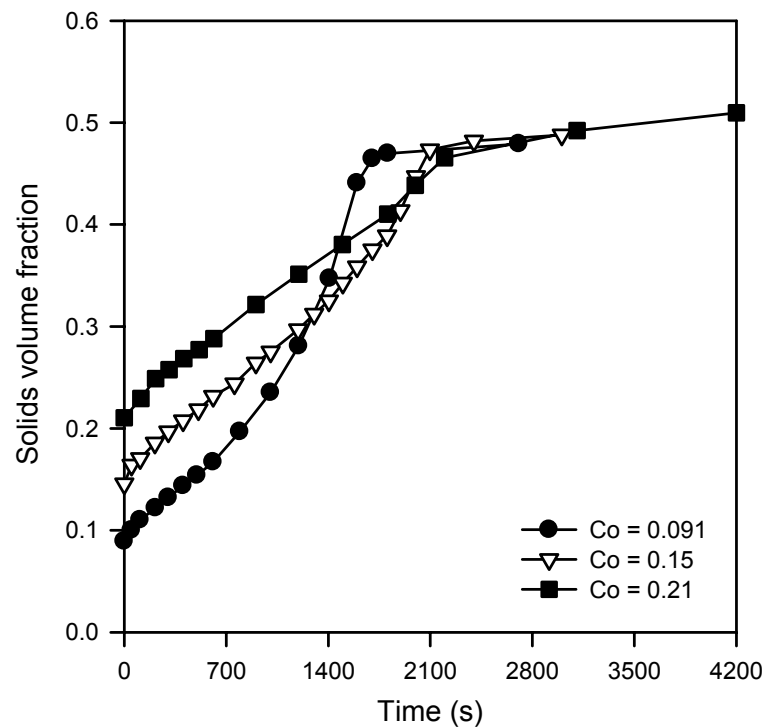


Figure 3: Expression of calcite at pH 11.8 and 31 bar.

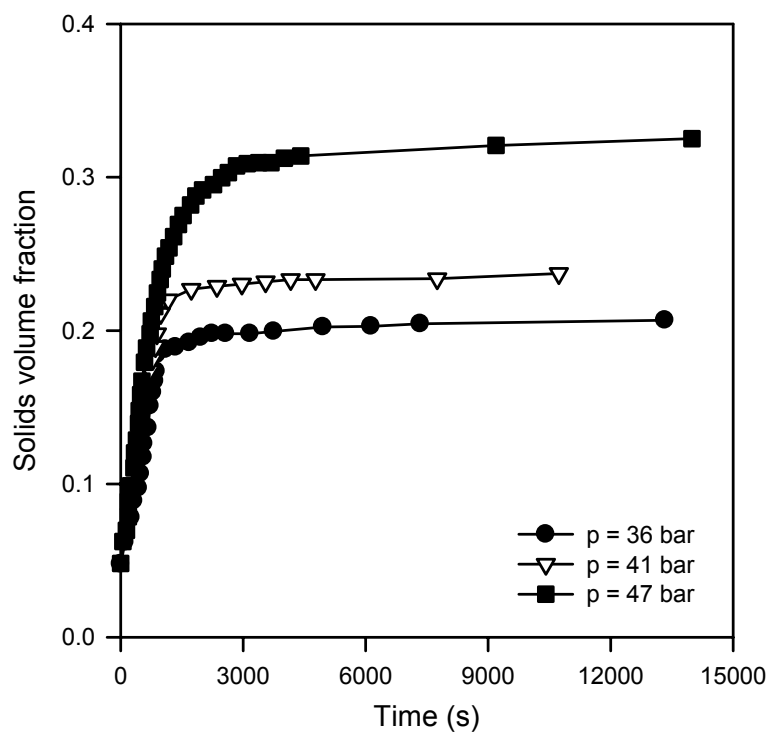


Figure 4: Expression of magnesium carbonate at pH 11.03.

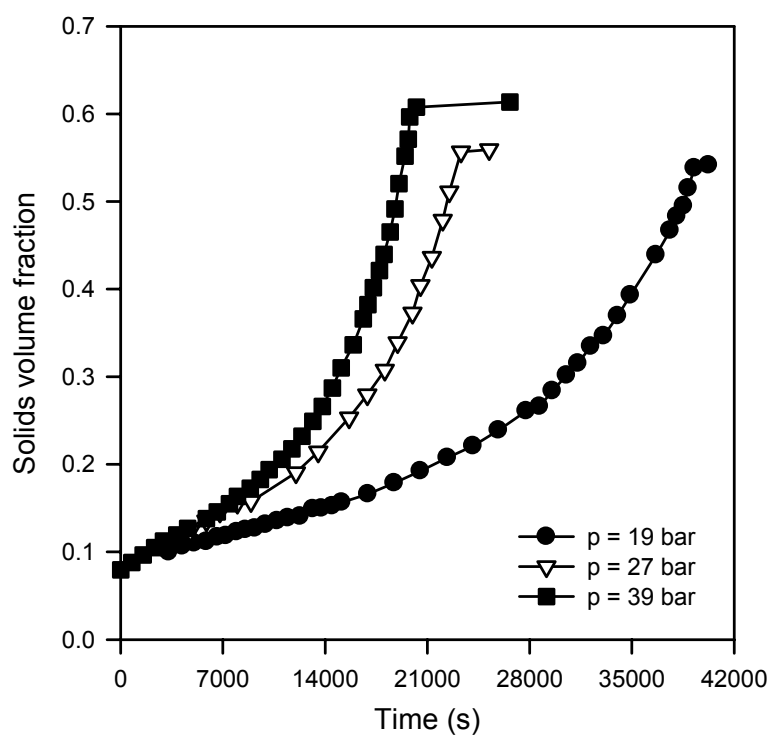


Figure 5: Expression of anatase at pH 10.22-10.36.

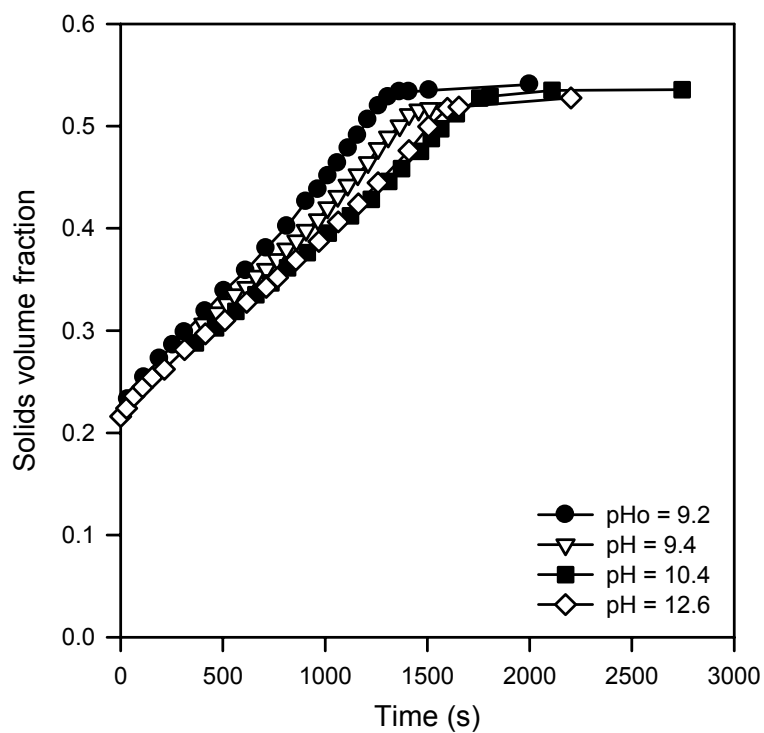


Figure 6: Expression of calcite at 28.8-32.5 bar.

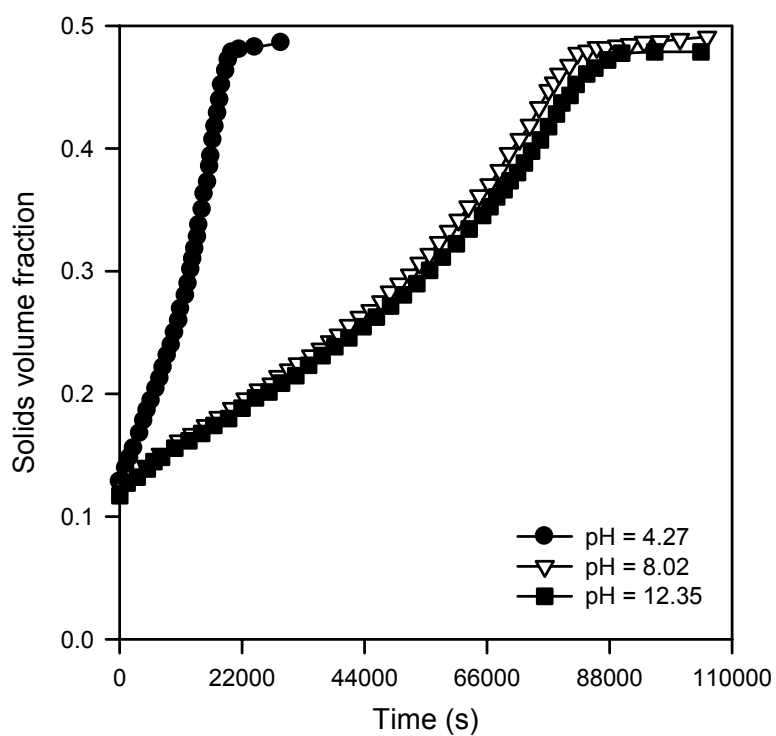


Figure 7: Expression of china clay at 35.3 bar.

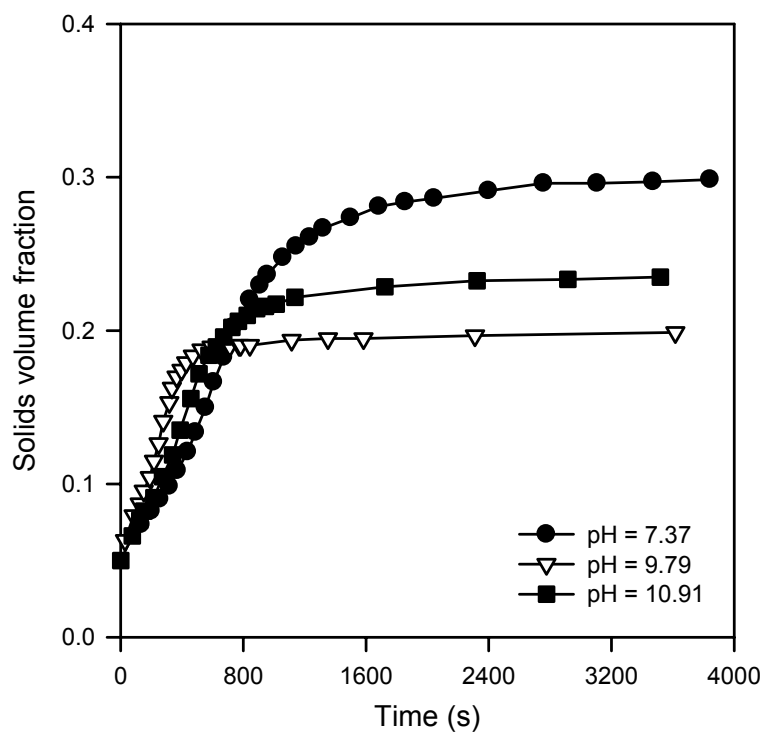


Figure 8: Expression of magnesium carbonate at 44.6 bar.

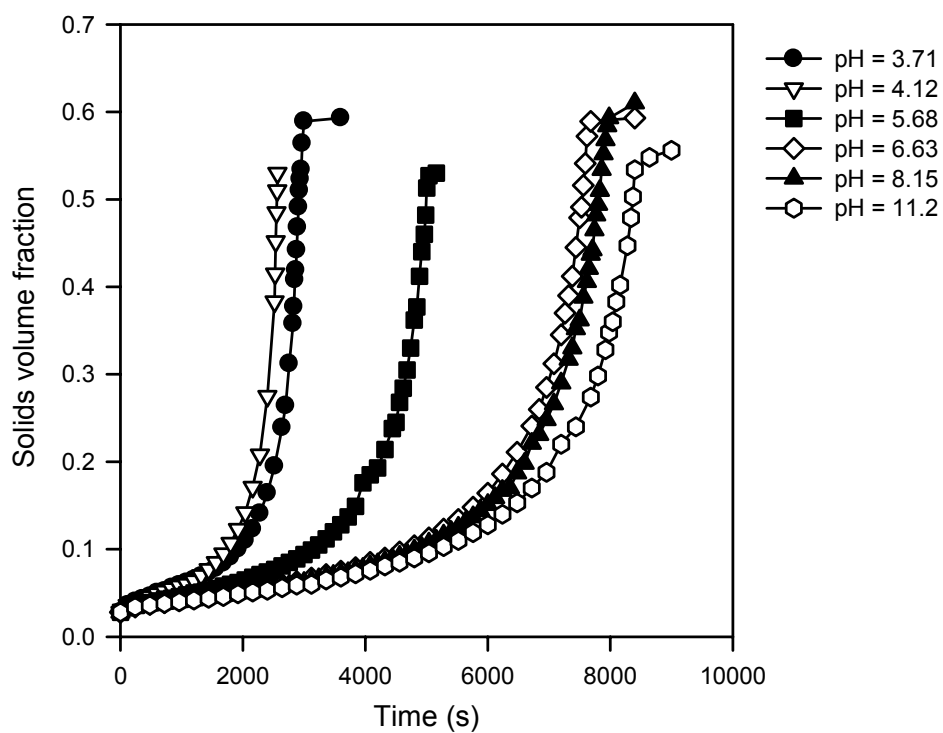


Figure 9: Expression of anatase at 43-46.4 bar.

Material	Particle size (μm)	Shape	Surface charge
Calcite (CaCO_3)	3 and 15	ca. Rhomboidal	Low
Anatase (TiO_2)	0.5	ca. Tetragonal	High
Kaolinite	0.45	Platelets	Medium-high
Hydromagnesite (MgCO_3)		ca. Rhomboidal	High

Table 1: Materials used.