

**ANALYSIS OF THE SYSTEM PRECIPITATION-SEPARATION UNIT.
EFFECT OF FILTRATION PRESSURE DROP ON RELATION AMONG
FILTRATION PROPERTIES OF SUSPENSIONS
AND REACTION CONDITIONS AT PRECIPITATION**

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Filtration characteristics of BaCO_3 and SrCO_3 suspensions precipitated batchwise at differing conditions were studied in dependence on the used filtration pressure drop. Both compounds form compressible filtration cakes whose behaviour is described by an exponential function. Parameters of this function determined experimentally for $\Delta P = 20$ to 80 kPa are given. Theoretical relation between initial supersaturation at precipitation and filtration resistance of the formed suspension (Eq. (14)) hold for both compounds in the whole studied range of filtration pressure drops ΔP from 20 to 80 kPa. Two groups of compounds were found; for the first one the parameters of Eq. (14) are not a function of ΔP , whereas for the second one the dependence of parameters in Eq. (14) on ΔP is significant.

In previous studies¹⁻⁶ the relation between filtration properties of precipitated suspensions and reaction conditions at their origin was verified. Filtration properties of suspensions were, however, in all cases determined always at one filtration pressure drop. The question arises whether the experimentally found constants of such dependences are independent of filtration pressure drop or whether they change significantly with ΔP .

The effect of filtration pressure drop (vacuum) on relation between filtration properties of precipitated suspensions and reaction conditions at precipitation is determined in this study using BaCO_3 and SrCO_3 as model compounds.

THEORETICAL

Filtration properties of suspension are characterised by the so called specific filtration resistance defined as

$$\alpha = f(\varepsilon) s^2 / \rho_s, \quad (1)$$

where $f(\varepsilon)$ is mostly expressed as

$$f(\varepsilon) = K_1(1 - \varepsilon)\varepsilon^3. \quad (2)$$

The constant K_1 becomes equal to 2 in the case when there is a system of parallel capillaries of identical dimensions in the filtration cake. When the cake is formed by randomly arranged particles K_1 is a function of particle shape and of bed porosity⁷. K_1 is thus not exactly constant, nevertheless for incompressible cakes it becomes equal to $K_1 = 5 \pm 0.5$. In the case of compressible cakes K_1 is variable. As it advantageous to keep $K_1 = 5$ the procedure proposed by Grace⁸ is adopted, where the specific surface area of the solid phase in Eq. (1) is substituted by the effective specific surface area s_{eff} , which depends on filtration conditions.

As porosity of the cake is not identical throughout its volume, it is advantageous to introduce the so called average bed porosity defined as

$$\varepsilon_{\text{av}} = 1 - G/h_c \rho_s A. \quad (3)$$

Eq. (1) then becomes

$$\alpha_{\text{av}} = 5(1 - \varepsilon_{\text{av}}) s_{\text{eff}}^2 / \rho_s \varepsilon_{\text{av}}^3. \quad (4)$$

The mean specific filtration resistance α_{av} is a function of filtration pressure drop and of concentration of the filtrated suspension^{8,9}, i.e.

$$\alpha_{\text{av}} = f(\Delta P, x_s). \quad (5)$$

From the experiments⁸ resulted that the dependence of α_{av} on ΔP can be in a limited range of pressures expressed by relation

$$\alpha_{\text{av}}(\Delta P) = \alpha_0(\Delta P)^{\beta'}. \quad (6)$$

Similarly, for the mean volumetric specific filtration resistance r_{av} , which is related with α_{av} by expression

$$r_{\text{av}} = \alpha_{\text{av}} \rho_s (1 - \varepsilon_{\text{av}}) \quad (7)$$

holds

$$r_{\text{av}}(\Delta P) = r_0(\Delta P)^{\beta}. \quad (8)$$

The dependence of average porosity of the cake on filtration pressure drop can be in a limited range of pressures expressed⁸ as

$$\varepsilon(\Delta P) = \varepsilon_0 - \gamma \log(\Delta P) \quad (9)$$

or¹⁰

$$\varepsilon(\Delta P) = \varepsilon_0(\Delta P)^{-\delta}, \quad (10)$$

where $\delta = 0.005$ to 0.05 . Besides these relations there were also proposed relations^{8,10,11}

$$\varepsilon_{av}/(1 - \varepsilon_{av}) = a_1 \ln \Delta P + a_2 \quad (11)$$

$$1/(1 - \varepsilon_{av}) = a_3 + b(\Delta P)^{\alpha} \quad (12)$$

$$(1 - \varepsilon_{av}) = a_4(\Delta P)^{\nu} \quad (13)$$

The dependence of ε_{av} on concentration of filtered suspension has not yet been expressed analytically. According to^{4,7}, ε_{av} decreases with increasing x_s and it could also pass through a maximum at a certain value x_s .

The mean volumetric specific filtration resistance of the batchwise precipitated suspensions depends on the initial supersaturation according to¹

$$y = Ex + F, \quad (14)$$

where

$$y = \log(r_{av} \cdot S^{2/3}) \quad (15)$$

$$x = (\log S)^{-2} \quad (16)$$

$$E = 2[C + Ah(n) - A]/3 \quad (17)$$

$$F = \log [(32F_t/F_{Re}) \cdot (\rho_s \pi / 2Mc_{eq})^{2/3} \cdot 10^Y] \quad (18)$$

and where

$$Y = (2/3) [D + \log g(n)/\tau^{(h(n)-1)} + (h(n) - 1)B + \log 3].$$

In relations (17) and (18) A , B , C and D are constants related to the kinetics of nucleation and $g(n)$ and $h(n)$ are functions of the kinetic nucleation order. In spite of the theoretical significance of constants E and F it is better to consider them as adjustable parameters as due to complexity of Eqs (17) and (18) even a small inaccuracy in numerical values of precipitation parameters significantly change the values of constants E and F .

EXPERIMENTAL

Batchwise precipitation has been performed in a beaker into which 200 ml of solution of BaCl_2 or SrCl_2 of known concentration at 25°C was introduced. Identical volume of the equimolar solution of Na_2CO_3 was quickly added to the chloride solution at intensive mixing by a blade stirrer. After 15 min of mixing 100 ml of suspension has been withdrawn for the filtration test.

All solutions prepared by dissolving the analar grade reagents in distilled water were left for 24 hrs to age after their preparation.

Filtration tests were performed on the standard testing equipment^{1,2} with the plate area 12.7 cm² at pressure drops 20, 40, 60 and 80 kPa. As filtration medium, filtration paper Filtrak 390 was used. The height of cake has been determined in the moment when the liquid surface has touched the cake surface. The cake was then washed by 50 ml of water and was dried to constant weight. For each filtration test new suspension has been prepared. Filtration properties of suspension prepared at given conditions were always determined twice with independently prepared suspension and the obtained results were averaged.

RESULTS AND DISCUSSION

Concentration of solid phase in suspension prepared by precipitation of solutions with concentration c_i has been calculated as

$$x_s = c_i M / 2. \quad (19)$$

Average porosities of the filtration cake determined on the basis of filtration tests according to Eq. (3) were correlated together with the data¹ as function of x_s using $\rho_s = 4\,430$ and $3\,740 \text{ kg m}^{-3}$ for BaCO_3 and SrCO_3 . Resulting relations which hold within the range of filtration pressure drops from 20 to 80 kPa are for BaCO_3

$$\varepsilon_{av} = -2.25 \cdot 10^{-4} x_s + 0.881 \quad 40 < x_s < 150 \quad (20)$$

and for SrCO_3

$$\varepsilon_{av} = -4.24 \cdot 10^{-4} x_s + 0.879 \quad 60 < x_s < 165. \quad (21)$$

Similarly as with the other studied systems², porosity of the filtration cake is also for BaCO_3 and SrCO_3 a function of initial supersaturation at precipitation. Solid phase formed by precipitation from the system with increasing initial supersaturation forms a filter cake having a decreasing average porosity.

Suspensions prepared at the given supersaturation were always filtered at pressure drops 20, 40, 60 and 80 kPa. As the suspensions of BaCO_3 and SrCO_3 were prepared at 4 different supersaturations, 16 values for each compound were obtained. From these results 2 types of dependences were obtained. The first type has described the dependence of filtration resistance of the suspension prepared at the given supersaturation on the given filtration pressure drop. For each compound four dependences $\alpha = f(\Delta P)$ were obtained where each holds for different supersaturation (Table I). The second type has expressed the dependence of filtration resistance on initial supersaturation at the given filtration pressure drop. For each studied compound 4 dependences $\alpha = f(S)$ were obtained, where each holds for different value of the used ΔP (Table II).

Dependence of specific filtration resistances of suspension r_{sv} and α_{sv} on filtration pressure drop within the range from 20 to 80 kPa (the first type of dependence) were for each used initial supersaturation of the system expressed by Eqs (7) and (8). The parameters of these dependences determined by the last square method are given in Table I. With regard to the fact that these parameters do not change significantly for the given compound, it is possible to consider the behaviour of the solid phase which has been prepared at different supersaturations (and thus has differing granulometric composition) as constant, what concerns compressibility of the cake. On com-

TABLE I
Parameters of Eqs (7) and (8) valid from $\Delta P = 20$ to 80 kPa

| Compound | <i>S</i> | $\log r_0$ | β | $\log \alpha_0$ | β' |
|-------------------|----------|------------|---------|-----------------|----------|
| BaCO ₃ | 348 | 13.072 | 0.416 | 10.250 | 0.449 |
| | 478 | 13.552 | 0.352 | 10.625 | 0.475 |
| | 577 | 13.744 | 0.350 | 10.948 | 0.370 |
| | 650 | 13.777 | 0.421 | 10.864 | 0.430 |
| SrCO ₃ | 1 346 | 12.748 | 0.619 | 10.398 | 0.443 |
| | 1 250 | 12.779 | 0.629 | 10.098 | 0.635 |
| | 1 136 | 13.135 | 0.338 | 10.434 | 0.340 |
| | 976 | 11.939 | 0.775 | 9.242 | 0.772 |

TABLE II
Parameters of Eq. (14)

| Compound | <i>S</i> | ΔP | $-E$ | <i>F</i> | <i>k</i> |
|-------------------|-----------|------------|--------|----------|----------|
| BaCO ₃ | 348–650 | 80 | 26.44 | 19.75 | 0.990 |
| | | 60 | 27.17 | 19.76 | 0.995 |
| | | 40 | 28.51 | 19.86 | 0.999 |
| | | 20 | 27.93 | 19.71 | 0.998 |
| SrCO ₃ | 976–1 346 | 80 | 52.53 | 21.42 | 0.959 |
| | | 60 | 88.76 | 25.13 | 0.952 |
| | | 40 | 70.87 | 23.12 | 0.973 |
| | | 20 | 107.70 | 27.01 | 0.987 |

bining Eqs (6) and (8) equation similar to Eq. (13) is obtained

$$(1 - \varepsilon_{av}) = (r_0/\rho_s \alpha_0) (\Delta P)^{\beta - \beta'}, \quad (22)$$

where the condition $(\beta - \beta') < 0$ must be met. As the results in Table I. mostly do not satisfy this condition, the determined parameters of Eqs (6) and (7) are affected by an experimental error covering the expected trend. Therefore these dependences should be considered as approximate empirical functions on basis of which it is not possible to make other conclusions than that BaCO_3 and SrCO_3 form compressible cakes filtration resistance of which increases with increasing filtration pressure drop in the studied range of ΔP according to the corresponding function.

Relation among filtration resistance of the precipitated suspension and conditions at precipitation expressed by Eq. (14) (second type of dependence) has been verified for both studied compounds (Table II). In the case of BaCO_3 both parameters of Eq. (14) are independent within the range of experimental inaccuracies on the used filtration pressure drop in the range from 20 to 80 kPa. For SrCO_3 the correlation by use Eq. (14) fits the experimental data less than for BaCO_3 . Moreover the numerical values of parameters in Eq. (14) change rather significantly in dependence on the used ΔP . Even if the values for $\Delta P = 80$ kPa are eliminated as clearly differing from the remaining values, the change in E and F at variable ΔP cannot be still ascribed only to experimental errors. In this case it must be accepted that parameters of Eq. (14) are for SrCO_3 dependent on the used filtration pressure drop.

From the present results and especially from Table II results that there exist two groups of compounds as concerns the effect of pressure on their filtration properties. In the first group (in our case BaCO_3) the filtration pressure does not have an effect on constants of Eq. (14) expressing the relation between the precipitation conditions and filtration properties of the formed suspension at least within the range from 20 to 80 kPa. In such a case it is immaterial which ΔP is used in the experiments as the calculated constants E and F in Eq. (14) are valid for any pressure. In the second group of compounds (here SrCO_3) are the determined constants E and F dependent on ΔP used in the experiments and it is not possible without a greater error to apply them for altered filtration conditions. At presentation of results concerning the relation between precipitation conditions and filtration properties it is necessary to state ΔP at which the experiments were performed in the case the compound belongs into the second group of compounds, *i.e.* into the group where the constants of Eq. (14) are a function of ΔP .

LIST OF SYMBOLS

| | |
|----------------------|-----------|
| A, B, C, D | constants |
| a_1, a_2, a_3, a_4 | constants |

| | | |
|-------------------------------|---|----------------------|
| A | area of the filtration element | m^2 |
| c | concentration of precipitated solutions | mol dm^{-3} |
| c_{eq} | equilibrium concentration | mol dm^{-3} |
| E, F | constant of Eq. (14) | |
| F_f, F_{Re} | friction factor and Reynolds factor | |
| $f(\varepsilon)$ | function of porosity | |
| G | mass of dry material of the cake | kg |
| h_c | cake height | m |
| k | correlation coefficient | |
| K_1 | Carman constant | |
| M | molecular mass | |
| ΔP | pressure drop | kPa |
| r | volumetric specific filtration resistance | m^{-2} |
| S | supersaturation | |
| s | specific surface area of the solid phase | m^{-1} |
| s_{eff} | effective specific surface area of the solid phase as concerns filtration | m^{-1} |
| x_s | concentration of suspension | kg m^{-3} |
| α | specific mass filtration resistance | kg m^{-1} |
| β, β' | exponents | |
| ε | bed porosity | |
| ρ_s | density of solid compound | kg m^{-3} |
| $\gamma, \delta, \omega, \nu$ | exponent | |
| τ | time | s |

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