Effect of viscosity in the porosity of granular materials in the moving bed washing process

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Efecto de viscosidad en la porosidad de materiales granulares en procesos de lavado en lecho móvil

Efecte de viscositat en la porositat de materials granulars en processos de rentat en llit mòbil

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RESUMEN

Los procesos de lavado se caracterizan por el reemplazo de los líquidos iniciales por otro líquido (el líquido de lavado). En el artículo que se expone, los estudios experimentales se han realizado en un sedimentador de laboratorio. Se expone un incremento en la porosidad del lecho móvil con la velocidad vertical media a un ancho fijo de la cámara de lavado. Las diferencias oscilan entre 3-5% para las condiciones elegidas. Los resultados experimentales muestran que los valores de porosidad en cama móvil, para ambas fracciones de arena, son ligeramente superiores a los valores del lecho móvil con relación de viscosidad (η *) igual a uno.

Palabras clave: Filtro; sedimentador; lavado; viscosidad.

SUMMARY

Washing processes are characterized by the replacement of the initial liquids by another liquid (the wash liquid). In the paper being presented, the experimental studies have been conducted in lab sedimentation equipment. It was exhibited an increment in the porosity of the moving bed with the average vertical velocity at a fixed width of the wash chamber. The experimental results show that the porosity values in the moving bed for both fractions of sand are slightly higher than the values of the moving bed with viscosity ratio (η*) equal to one. The differences are in the range of 3-5 % at the chosen conditions.

Keywords: Filter; sedimenter; washing; viscosity.

RESUM

Els processos de rentat es caracteritzen pel reemplaçament dels líquids inicials per un altre líquid (el líquid de rentat). A l’article que es presenta, els estudis experimentals s’han fet en un sedimentador de laboratori. S’exposa un increment en la porositat del llit mòbil amb la velocitat vertical mitjana a un amplada fixa de la càmera de rentat. Les diferències oscil·len entre 3-5% per a les condicions escollides. Els resultats experimentals mostren que els valors de porositat en un llit mòbil, per a les dues fraccions de sorra, són lleugerament superiors als valors del llit mòbil amb relació de viscositat (η*) igual a un.

Mots clau: Filtre; sedimentador; rentat; viscositat.

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1. INTRODUCTION

Washing processes are extended in various industrial applications and they are of great importance. Those operations are mainly focussed on the separation of valuable or undesired liquids or therein dissolved substances from a system of solid particles. These processes are characterized by the replacement of the initial liquids by another liquid (the wash liquid) [**Franky et al. 2009**]. Both liquids do not have to be necessarily miscible; the paper being presented is limited only to miscible liquids. There are several ways to carry out an efficient washing process; there are distinguished two basic procedures: washing by dilution and cake washing.
The separation of fluids containing soluble substances from sediments and filter cakes are one of the most important operations for various chemical processes either to treat raw materials or to purify products [**Choua et al 2014**]. The substances dissolved are present not only in the liquid that can be moved between the particles, but can also be located within the pores of more porous particles or at the boundary surface of liquid-solid phase.
Hoffner and colleagues worked several years in the study of washing process of quartz sand in the moving bed [**Hoffner et al. 2001, 2003 and 2005**]. The concept for a new washing process using a moving bed aims to combine advantageous characteristics of two well-known washing processes: displacement of mother liquor as the dominant transport mechanism avoiding disadvantages of a fixed bed structure (e.g. impenetrable regions and channelling) and the interaction with boundaries (e.g. maldistribution of the wash liquid feed, maldistribution of mother liquor due to a filter cloth and its support). The washing process described here is based on two requirements: first, a density difference between two solids and liquids; second, the solids form a free flowing bulk when immersed in the respective fluids/solutions. These requirements are fulfilled for various particulate products.
The moving bed shows bulk flow behaviour. It differs from the fixed bed structure due to the relative movement of the particles. The porosity of the moving bed increases with the average velocity of the moving bed at a fixed width of the wash chamber. This corresponds to observations of flowing, bulk materials **[Rodríguez-Machín 2008; Hoffner et al. 2003]**. The measured values for the porosity are all higher than the values of a corresponding filter cake ($\Delta p <$ 0.2 bar), but not necessarily higher than the ones of gently formed sediments. In 2004, Hoffner and Stahl demonstrated that the relative movements of particles leads to a slight porosity increase compared to the porosity of a fixed bed. The porosity increase measured under these conditions ($\eta^*=1$) was in the range of 2-3 % absolute and is still in the range of a gently formed sediment.
Mineral building materials are among the most important materials in the construction industry. Quartz sand is used as a major component in industrial flooring but also opens up the full range of potential applications, ranging from indoor/outdoor plaster and render through polymer concrete and dry mortar to applications in construction chemistry. Therefore, the quartz sands demands high quality. The porosity of the sand is an important consideration when attempting to evaluate the potential volume of liquid it may contain or it is necessary to remove. Also in washing process, porosity measurements are needed to get information about the moving bed structure. The washing of quartz sand in moving bed is especially affected by the material properties, particularly the viscosity of the liquids used. The objective is to study the effect of viscosity in the porosity of granular materials in the moving bed washing process.

2. MATERIALS AND METHODS

2.1 Experimental setup

The experimental studies were conducted with a plain, single-stage lab Sedimenter described in Figure 1. Fully saturated sediment enters the wash chamber continuously from the top.

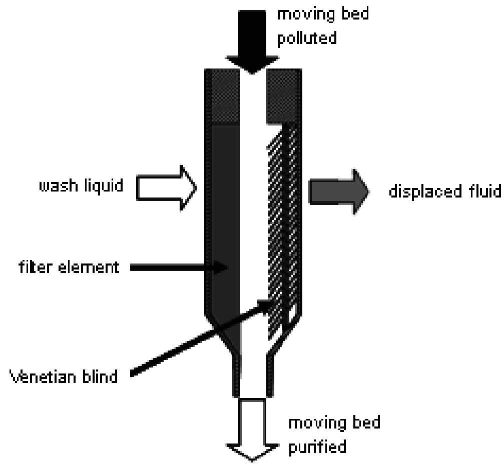

Figure 1. *Single wash chamber of plane lab-Sedimenter.*

The chamber consists of a filter element at the wash liquid entry and a "Venetian blind" type fitting at the mother liquor exit, a rear wall, and a glass front plate in order to observe the movement of solids and liquids. These boundaries form a rectangular channel for the moving bed. Solids and mother liquor enter on top. Having entered the wash chamber the moving bed is penetrated by the wash liquid and the mother liquor is displaced. Both fluids are pumped volumetrically with a double headed low pulsation peristaltic pump into and out of the wash chamber. The purified moving bed exits to a screw conveyor that transports the washed moving bed to the Sedimenter. In order to avoid an undesired vertical relative movement of the pore liquids with respect to the solids, three pressure indicators are placed between the exit of the wash chamber and the screw conveyor in this way the pressure drop is monitored along the device in order to ensure that the values corresponds to the expected hydrostatic pressure difference.
The use of filter media at the wash liquid exit in the wash chamber could cause serious disadvantages, e.g. blocking, wear, and a random distribution of the fluids due to impenetrable areas of the filter cloth and its support. Using a "Venetian blind" shaped fitting in order to guide the moving bed can reduce or even avoid the points mentioned. Additionally, the wash chamber can be depleted without any residues in the case of a product change.
The residual impurity content is obtained by a conductivity measurement at the inlet section of the screw conveyor where a small amount of interstitial water is continuously removed and recycled further up the screw conveyor. The salt concentration was monitored and used for the calcu-

lations only after it had been stationary for at least 30 minutes **[Rodríguez-Machín 2008; Hoffner 2001]**.

2.2 Theoretical description of the washing process

In a washing process there are two important parameters: the wash ratio (W) and the remaining fraction of impurity (X*).

2.2.1 Wash ratio

$$W = \frac{V_w}{V_p} \quad (1)$$

VW = the volume of wash liquid (m³)
Vp = the pore volume (m³). The wash ratio is always ≥ 0.

2.2.2 Remaining fraction of impurity

$$X^* = \frac{C_1 - C_w}{C_0 - C_w} \quad (2)$$

Here:
ε = the porosity (-) is constant at 0 ≤ X* ≤ 1
c_0 = the salt concentration of the total entering moving bed (g/l)
c_1 = the salt concentration of the total leaving moving bed (g/l)
c_W = the salt concentration of the wash liquid (g/l)
In the ranges explored the conductivity measurements exhibited a weak dependency on the temperature and on the concentration of the viscosity-increasing additive, both expressed with the coefficients, k_{TL} and k_{KL} as determined by **Rodríguez - Machín 2008** and integrated in Eq. (3). Therefore, to calculate the concentration of NaCl Eq. (4) was used.

$$\kappa(T, C_{PVP}) = \kappa_0 (1 + k_{TL}(T - T_0))(1 + k_{KL} c_{PVP}) \quad (3)$$

κ_0 = to κ when the temperature is 25°C and c_{PVP}=0
k_{TL}= temperature coefficient
k_{KL} = concentration coefficient
κ_0 = the measured value of the conductivity

$$\kappa(T, C_{PVP}) = \kappa_0 (1 + 2{,}07(T - T_0))(1 - 0{,}22 C_{PVP}) \quad (4)$$

In order to characterize the viscosity difference between the wash liquid and the mother liquor, the viscosity ratio (η*) is defined.

$$\eta^* = \frac{\eta_{wash\ liquid}}{\eta_{mother\ liquor}} \quad (5)$$

$\eta_{wash\ liquid}$ = the viscosity of the wash liquid (mPa s)
$\eta_{mother\ liquor}$ = the viscosity of the mother liquor (mPa s)
To get information about the moving bed structure, porosity measurements are needed. A sampling point was placed at the discharge of the screw conveyor in order to determine the porosity at different times. The wet mass and dry mass were determined thermogravimetrically **[Hoffner 2003; Hoffner 2005]**.

$$\varepsilon = \frac{V_l}{V_{tot}} = 1 - \frac{V_s}{V_{tot}} = 1 - \frac{m_s/\rho_s}{m_l/\rho_l + m_s/\rho_s} \quad (6)$$

Here:
V_l = volume of the fluid (m³)
V_{tot} = total volume (m³)
V_s = volume of the solid (m³)
m_s = mass of the solid (kg)
ρ_s = density of the solid (kg m⁻³)
m_l = mass of the fluid (kg)
ρ_l = density of the fluid (kg m⁻³)
In order to minimize the wash liquid demand, the impure liquid has to be displaced to a great extent avoiding a mixing with wash liquid and especially a bypass flow of wash liquid. The up to now investigations for the purification of quartz sand in a Sedimenter showed very good wash results, as much in reference to the remaining impurity as to the necessary specific washing water volumes. Thereby in the research presented here, mother and wash fluids with negligible viscosity differences were always used. From the process engineering literature and a preceding work about filter cake washing it is well-known, that larger or else different viscosities of the washing liquid in relation with the mother liquor can decrease or favour the tendency to the formation of preferential flow channels (viscous fingering) **[Bender 1983; Heuser 2003]**.

2.2.3 Course of the wash curve

The washing results are usually described by a washing curve where the remaining fraction of impurity (X*) is plotted versus the wash ratio (W). The typical washing curve can be divided into three areas as shown in Figure 2.

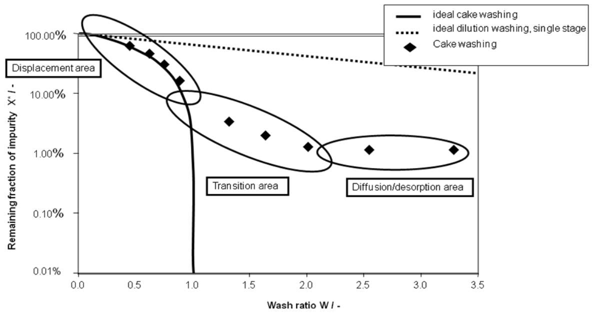

Figure 2. *Remaining fraction of impurity (X*) plotted versus the wash ratio (W).*

- <u>*Displacement area:*</u> In this area, the condition of the ideal displacement applies with the addition of small quantities of washing liquid. Diffusion and desorption effects play a negligible role.
- <u>*Transition area:*</u> Additionally there is a flow induced mixture (dispersion) between the mother and wash fluids which can be caused by inhomogeneity of the pore structure. The deviation from the ideal curve, the pistons shaped displacement, begin in this area.
- <u>*Diffusion/desorption area:*</u> In this area the impurities are transported by diffusion/desorption mechanism into the wash liquid. The remaining fraction of impurity reaches its limit value and the remainder pollution cannot be removed substantially by increasing the amount of the wash liquid **[Heuser 2003]**.

2.3 Washing modelling

Figure 3 shows two fractions of quartz sand used in the experiments being presented ($x_{50.3}$ = 337 µm (Dorsilit 9H) and 160 µm (Geba), ρ_s= 2650 kg m⁻³). Sodium chloride was used as impurity and therefore is the target substance to be removed during the washing process; which was dissolved in the mother liquor in a concentration of c_{NaCl} ≈12 g/l. The sodium chloride solution was prepared with demineralised water (conductivity of 0.8 - 9 µS/cm) and pure sodium chloride. Polyvinylpyrrolidone (PVP) was used as viscosity-increasing additive. Both the viscosity and density deviation of the mother liquor are negligible at room temperature (20 -30 °C).

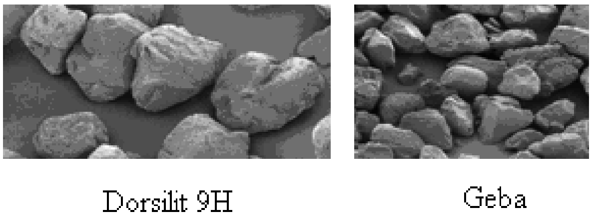

Figure 3. *Fractions of quartz sand used in the course of the experiments.*

3. RESULTS AND DISCUSSION

Hoffner (2005) have also demonstrated that the relative movement of the particles in the moving bed leads to a slight porosity increase compared to the porosity of a fixed bed, specifically the case of the respective filter cakes. On the other hand, it does not show a suspension-like behavior as flow experiments through a discharge gap below the wash chamber. The porosity is increased under these conditions in the range of 2 - 3 % in respect to the filter cakes formed at fixed bed washing experiments (Table 1). Gently formed sediments show porosity values in the same range as the moving bed.

Table 1. Porosity values of a moving bed and a filter cake and the respective relative standard deviation with η*= 1 **[Hoffner 2005]**.

	moving bed	filter cake (Δp< 0.2 bar)
Dorsilit 9H sand	47.2 %± 0.6%	44.6 %± 1%
Geba sand	46.3 %± 2.4%	45.7 %± 1%

The porosity of the bed in the wash chamber is not constant but increases with the average velocity of the moving bed, and therefore with the shear rate in the shear zone, see Figure 4.

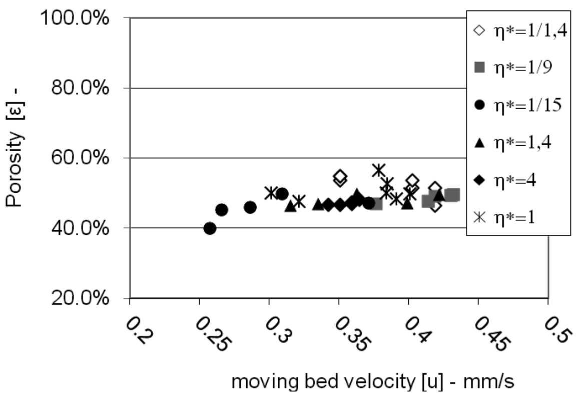

Figure 4. *Porosity of the moving bed as a fraction of the vertical moving bed velocity (sand Dorsilit 9H).*

In the case of the materials and conditions considered here, it grows by a few percentage points. The experimental results in this work show that the porosity values in the moving bed with the viscosity ratio ranging from 4 to 1/15 are slightly higher than the values of the moving bed with η*= 1. The differences are in the range of 3 - 5 %. The course of porosity values for sand Geba shows qualitatively the same behavior as sand Dorsilit, see Figure 5.

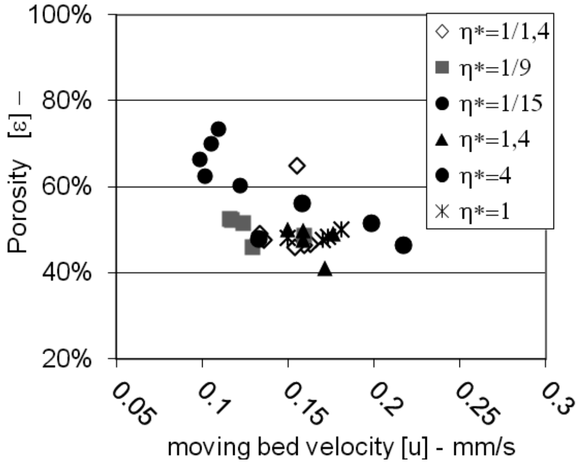

Figure 5. *Porosity of the moving bed as a fraction of the vertical moving bed velocity (sand Geba).*

In the case of the sand fraction with small size particles the increase in the porosity is higher due to the same volume of bed there is a greater number of particles causing an increase in empty spaces between them. The velocity of the fluid through the moving bed in the wash chamber undergoes an increase with decreasing porosity of the particles [**Sandorini and Franzoni 2001**]. The number of particles and therefore the interface area are higher when the size of particles in the same bed volume is smaller.

The results of investigations for the washing of the moving bed are covered with systematic deviations [**Sandorini and Franzoni 2001**]. The most important error sources are mentioned and quantified. First and foremost is the impact of systematic errors conveyors and concentration determination. In addition, also the errors due to the uncertainty in the determination of the adsorption isotherm influence **[Isshi et al. 2009]**. The following deviations of target and actual values were determined for respectively, assessed and introduced into a maximum error bill,

Washing fluid pump: 1.5%

Screw conveyor speed: 2%

Volume flow of the pressure compensation pump: 3%

Error of the conductivity measurement (mother liquor, entry): 1%

Error of the conductivity measurement (wash liquid, entry): 5%

Error of the conductivity measurement (mother liquor, exit): 5%

In addition there are other possible errors which are however difficult to quantify.

4. CONCLUSIONS

The experimental results of the washing processes used showed similar tendencies for both sand fractions when there are viscosity variations of mother liquor or wash liquid. The differences in respect to the values of the moving bed with η*= 1 are in the range of 3 - 5 % at the chosen conditions. The velocity of the fluid through the moving bed in the wash chamber undergoes an increase with decreasing porosity of the particles. The number of particles and therefore the interface area are higher when the size of particles in the same bed volume is smaller.

5. ACKNOWLEDGEMENTS

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