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Mechanical Dewatering of Homogeneous and Segregated Filter Cakes by Vibration Compaction

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Abstract: The solid volume fraction of a slurry requiring solid–liquid separation often fluctuates in industrial cake filtration processes. For low solid volume fractions, particle segregation arises, resulting in an inhomogeneous filter cake structure. Particle segregation has significant impacts on cake formation such as a longer cake formation time compared to homogeneous cakes. This work addresses the impact of this effect on vibration compaction, which is an alternative deliquoring method applying oscillatory shears to the filter cake. The dewatering results of homogeneous and segregated cakes made of the same material with a broad particle size distribution are compared. Although cake deliquoring is achievable despite particle segregation, vibration compaction is more effective for homogeneous cakes. The reason is that no particle size homogenization within segregated cakes occurs due to oscillatory shear, as particle size analyses indicate. The particle size measurements of cakes before and after vibration compaction reveal that the material's particle size distribution is preserved despite vibration application. Vibration compaction achieves higher deliquoring than the common compaction method by squeezing, as elastic recovery effects after squeezing lead to the reabsorbing of liquid, already expressed and stored in the filter cloth. This demonstrates that vibration compaction is a real alternative for cake deliquoring.

Keywords: cake filtration; particle segregation; mechanical deliquoring; compressible filter cakes; vibration compaction

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1. Introduction

To separate solid particles from a liquid phase, mechanical processes are used as much as possible due to their significantly lower energy consumption than thermal drying [1–3]. Cake filtration is a mechanical separation method, especially for concentrated slurries in various sectors such as the mining industry [4,5], the food industry [6,7], or wastewater treatment [8,9]. By supplying the slurry onto a filter medium and applying a driving force such as a pressure difference, a porous filter cake is formed on the medium from deposited particles with residual liquid in the void volume. Mechanical methods exist for removing further liquid from filter cakes, such as the application of a gas pressure difference, blowing pore liquid out of the cake, or the compaction of compressible filter cakes, removing liquid by reducing pore volume [10].

The initial solid volume fraction of the slurry has a considerable influence on cake filtration. To achieve cake formation on the filter medium at all, to prevent clogging of the filter medium due to particle deposition inside the medium, and to minimize particle penetration through the medium, a sufficiently high solid volume fraction of the slurry should be ensured. The cake formation time decreases at a constant cake height with increasing solid volume fraction, resulting in a higher solid mass throughput of the filter apparatus [11,12]. Another reason to provide a high solid volume fraction of the slurry is particle segregation, which is a common problem in cake filtration, especially for particle materials with a broad particle size distribution [11,13–16]. Superimposed particle sedimentation appears during the cake formation process, depending significantly on the solid volume fraction of the slurry. If the solid volume fraction is too low, the particles settle at different sedimentation velocities according to their particle size during cake formation, resulting in a stratification of the particles in the filter cake. Coarse particles are located in the bottom area of the formed filter cake near the filter medium, while finer particles are found in the upper cake layers. At a sufficiently high solid volume fraction, mutual particle hindering occurs, whereby the entire particles settle in a sharp front with an equal sedimentation velocity. Consequently, the individual particle size loses its influence on the particle sedimentation velocity. This creates a homogeneous cake structure where the particles with different sizes are evenly distributed across the cake height.

Particle segregation during cake formation leads to a higher residual cake moisture compared to homogeneously formed filter cakes, as voids between coarse particles are less filled by smaller particles, and thus a higher porosity exists in segregated filter cakes [11,13,16]. In addition to the negative consequences for cake formation, there are problems with following process steps such as washing or post-deliquoring of segregated filter cakes. A layer of very fine particles on the filter cake surface impairs cake washing due to a higher flow resistance compared to homogeneous filter cakes [14]. Moreover, the fine particle layer raises the capillary entry pressure, which must be exceeded by a gas pressure difference to displace more pore liquid. In addition, the capillary entry pressure can only be exceeded locally in the fine layer. As a consequence, the liquid is only displaced in coarse pores, while small pores formed from fine particles on the cake surface remain filled with liquid. The result is uneven dewatering. In the worst case, the capillary entry pressure is greater than the maximum applicable pressure difference, which is strictly limited in the widely used vacuum filtration method, leading to no mechanical desaturation at all [11].

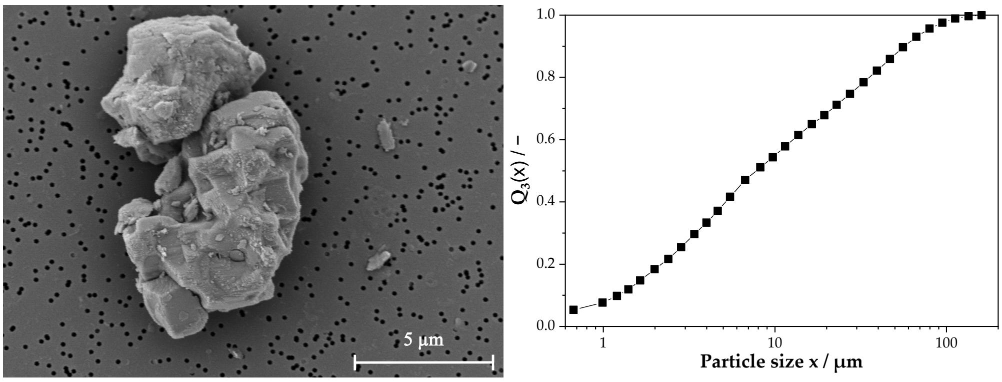
For fine-particle materials forming highly porous filter cakes with a porosity gradient across the cake height, filter cake compaction is another mechanical dewatering method [17]. An external compressive pressure triggers a particle rearrangement in the cake by destroying particle bridges and by elastic or plastic particle deformation, reducing the void volume and thus displacing more liquid from the cake [18–21]. The compressive pressure for noticeable cake deliquoring is often very high [21]. Squeezing the cake is performed in industrial cake filtration apparatuses by feeding further slurry under high pressure as in filter presses or by additionally installed mechanical compression devices such as press belts, press membranes, or inflatable pressure pads on belt filters [22–25]. The preferred cake filtration devices is the conventional belt filter, as it has a simple design and can achieve high throughputs due to continuous operation. For fine materials, the limited pressure difference generated by a vacuum is not sufficient to remove enough liquid, which is why discontinuous filter presses are mainly used in this case [26,27]. Although additional compression devices on belt filters improve deliquoring, the belt filter requires an extra design for such high loads, requiring costly constructive modifications, which deviate tremendously from the conventional, simple design of the belt filter [22]. However, there are also modular devices for additional deliquoring on existing belt filters that have a standard design, such as the add-on flapper roller according to Bickert and Vince [28] or rollers vibrating perpendicular to the filter cake surface according to Whatnall et al. [29]. An alternative method for mechanical cake dewatering is compaction by applying oscillatory shear parallel to the filter cake surface at a significantly lower compressive pressure. After Illies et al. [30–32] successfully demonstrated the process's potential for the mechanical deliquoring of mineral filter cakes on a discontinuous laboratory scale, Yildiz et al. [33] showed the feasibility of the process on an existing indexing belt filter at the pilot scale by using a developed vibration module. These investigations on vibration compaction were only based on homogeneously formed filter cakes. However, temporal fluctuations in the slurry solid volume fraction are possible in industrial filtration processes despite a thickener being used before the filtration step [34]. As no slurry homogenization during filtration is possible with belt filters, these devices in particular tend to form segregated filter cakes if the slurry concentration is too low and the material has a broad particle size distribution [35].

Therefore, this paper focuses on the impact of particle segregation during cake formation on the subsequent deliquoring by compaction under oscillatory shear to further understand the process and its applicability. For this purpose, the deliquoring and compaction results of homogeneous and segregated filter cakes were compared on the basis of experiments with the laboratory apparatus developed by Illies et al. [30]. Particle size measurements were intended to verify the preservation of the particle size distribution after vibration compaction and to characterize particle rearrangement as the main mechanism of vibration compaction. Investigating the possible particle penetration through the filter cloth as a result of vibration application to the cake was also part of the study. Compaction by squeezing as a common deliquoring method is the benchmark for vibration compaction as an alternative method. Thus, tests were carried out with a compression–permeability cell (CP cell) to compare vibration compaction with compaction by squeezing. Compaction and deliquoring results obtained by squeezing have only been compared with the results of vibration compaction during the load so far [30,31,33,36]. As the compressive pressure is obviously removed from the cake after a certain period on industrial belt filters, this time, the compaction was examined after squeezing, taking the elastic recovery effects of the cake into account.

2. Materials and Methods

2.1. Model Particle Material

The model particle material used for the study was a ground calcium carbonate material with a solid density of 2700 kg/m³. The particles had an angular, irregular shape (see Figure 1a). The particle size distribution of the material in deionized water measured with a HELOS H0309 laser diffraction instrument is shown in Figure 1b.



(a) **(b)**

Figure 1. (**a**) Scanning electron microscope image of the model particles and (**b**) cumulative particle size distribution of the model material measured in deionized water with HELOS H0309 laser diffraction instrument (Sympatec GmbH, Germany).

The mean particle size $x_{50,3}$ of the material was 8.6 µm. The particle size of the material was very broadly distributed, evidenced by the high *span* value of 7.1, which is the difference between $x_{90,3}$ and $x_{10,3}$, related to $x_{50,3}$, as a measure of the distribution width of a particulate material. Due to the widely distributed particle size, this material tends to show particle segregation during cake formation for small solid volume fractions of the slurry. Following Yildiz et al. [36], who reported correlations between the mean particle size and particle size distribution width of ground calcium carbonates and the vibration compaction behavior, the material is expected to be compressible by oscillatory shear. For these reasons, the choice of this material as a model particle material for this study was justified. The slurry samples were prepared by stirring the particles in deionized water with an RCT basic magnetic stirrer (IKA Werke GmbH & Co., KG, Staufen im Breisgau, Germany). As 40% was selected as the slurry solid volume fraction c_v for homogeneously formed filter cakes, the solid volume fraction c_v for segregated filter cakes was 10%. The solid mass was the same for both slurry samples and was chosen to provide a filter cake height of approx. 8 mm after cake formation for the slurry with 40%. The solid volume fraction was adjusted by the volume of water added.

2.2. Vibration Compaction of Filter Cakes

To analyze filter cake compaction by oscillatory shear as a mechanical deliquoring method, a discontinuous apparatus at the laboratory scale was utilized. The setup of the apparatus can be seen in Figure 2. The setup and the generation of the oscillatory shear are described in more detail in Illies et al. [30], who developed the apparatus.

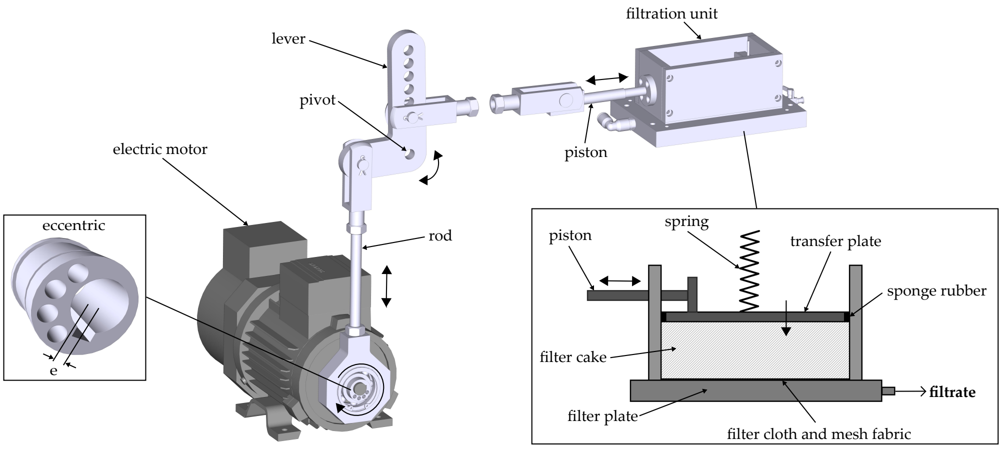


Figure 2. Apparatus developed by Illies et al. [30] for filter cake compaction by oscillatory shear at a low compressive pressure at a laboratory scale. The figure is from Yildiz et al. [33], reprinted with the permission of the publisher (Taylor & Francis Ltd., http://www.tandfonline.com, accessed on 23 Januar 2024).

By filling the slurry sample into the filtration unit and applying a pressure difference of 80 kPa by a vacuum in the filter plate, a filter cake was formed. Then, a plate was placed on the cake surface and applied an oscillatory shear with a constant shear length of 4.5 mm to the cake. Vibration compaction of homogeneous and segregated filter cakes was investigated at the highest frequency of 40 Hz that can be applied by the apparatus and at a moderate frequency of 17 Hz with a varying number of oscillations. A low compressive pressure of approx. 3 kPa generated by the spring force on the transfer plate and the weight of the transfer plate acted on the cake during vibration compaction, ensuring a defined contact between transfer plate and cake. Cake compaction by the low compressive pressure could be neglected. A pressure difference of 80 kPa was applied by a vacuum in the filter plate during vibration compaction to remove the liquid displaced by oscillatory shear below the filter medium. The vacuum in the filter plate did not contribute to the compressive pressure, as the plate and the sponge rubber attached around the plate did not completely cover the filter cake edge. A monofilament nylon filter cloth SEFAR NITEX© 03/5-1 (SEFAR AG, Thal, Switzerland) with a mesh size of 5 µm served as the filter cloth on the filter plate. Underneath the filter cloth was a stainless steel support mesh with a size of 1 mm (GKD-Gebr. Kufferath AG, Düren, Germany). SEFAR NITEX 03/5-1, also covering the transfer plate, allowed residue-free filter cake detachment from the plate after the removal of the plate.

2.3. Compaction of Filter Cakes by Squeezing

Using the compression–permeability cell (CP cell) shown in Figure 3 developed by Alles [37] made it possible to imitate the compaction of a previously formed filter cake using a squeezing device on a belt filter as a benchmark for the alternative vibration compaction method. After adding the slurry sample to the cake formation unit with a filter area A of 51.5 cm², cake formation took place first. Similar to the vibration apparatus (see Section 2.2), a pressure difference of 80 kPa for cake formation was applied with a vacuum in the filtrate drain bottom. A perforated stainless steel plate with a hole size of 240 µm, equipped with the same filter cloth SEFAR NITEX© 03/5-1 (SEFAR AG, Thal, Switzerland) as in the vibration apparatus, was placed on the filtrate drain bottom. A polypropylene support fabric with a pore size of 54 µm (SEFAR AG, Thal, Switzerland) was positioned between the perforated plate and the filter cloth. The formed cake was then squeezed with a piston. During squeezing, a stainless steel perforated plate with a hole size of 240 µm was between the piston and the filter cake, covered by polypropylene support fabric with a pore size of 54 µm and with a SUPOR© 100 hydrophilic polyethersulfone membrane with a pore size of 0.1 µm (Pall Corporation, Port Washington, WI, USA) instead of a filter cloth. The pressures tested were 100, 480, and 930 kPa, which were generated with a pneumatic cylinder connected to the piston. Squeezing of the cake lasted 120 s. As the cake height did not change significantly after this time in any of the cases, compaction equilibrium could be assumed. The filter cake height $h_{c,load}$ was derived from the continuous measurement of the piston position with a TLH 100 electrical position sensor (Novotechnik Messwertaufnehmer OHG, Ostfildern, Germany). A vacuum was also applied to the filtrate drain bottom with a pressure difference of 80 kPa during squeezing to drain liquid squeezed from the cake downward in the direction of the filtrate drain bottom. After the squeezing time had elapsed, the piston moved upwards and the filtrate drain bottom was ventilated.

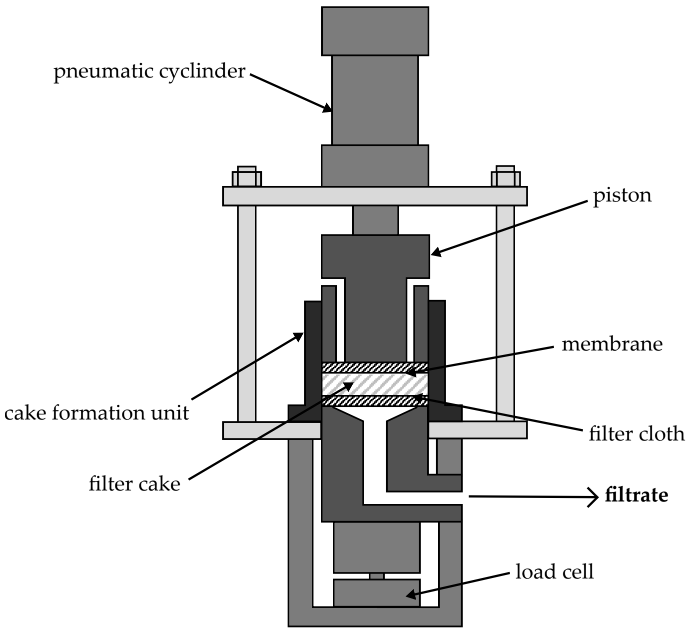


Figure 3. Setup of the compression–permeability cell developed by Alles [37]. The figure was taken from Yildiz et al. [33], reprinted with the permission of the publisher (Taylor & Francis Ltd., http://www.tandfonline.com, accessed on 23 January 2024).

2.4. Characterization of Filter Cake Properties

To characterize the compaction or deliquoring state of the filter cakes and the particle size distribution within the cake after formation or vibration compaction, we took cake samples from the filter plate using a cylindrical sampler with a diameter of 12 mm (see Figure 4a). The laser distance sensor LK-GK157 (KEYENCE Deutschland GmbH, Neu-Isenburg, Germany) first determined the total sample cake height h_c. Cutting the filter cake sample into several layers and then measuring the particle size distribution of the layers enabled the analysis of the particle size distribution within the filter cake. The developed device shown in Figure 4b, based on that of Anlauf [38], was used to cut filter cake samples taken from the filter plate of the vibration apparatus. After the sampler was clamped into the device with screws, a piston driven by a hand wheel pushed the filter cake out of the sampler in defined layer thicknesses, also measured with a laser distance sensor LK-GK157 (KEYENCE Deutschland GmbH, Neu-Isenburg, Germany). The layers cut out of the filter cake samples with a scalpel with a minimum layer thickness of up to 1 mm were analyzed in terms of particle size distribution with a HELOS H0309 laser diffraction instrument (Sympatec GmbH, Clausthal-Zellerfeld, Germany). The layer sample was suspended in deionized water and experienced an ultrasonic treatment for 2 min to disperse the particles before the particle size measurement.

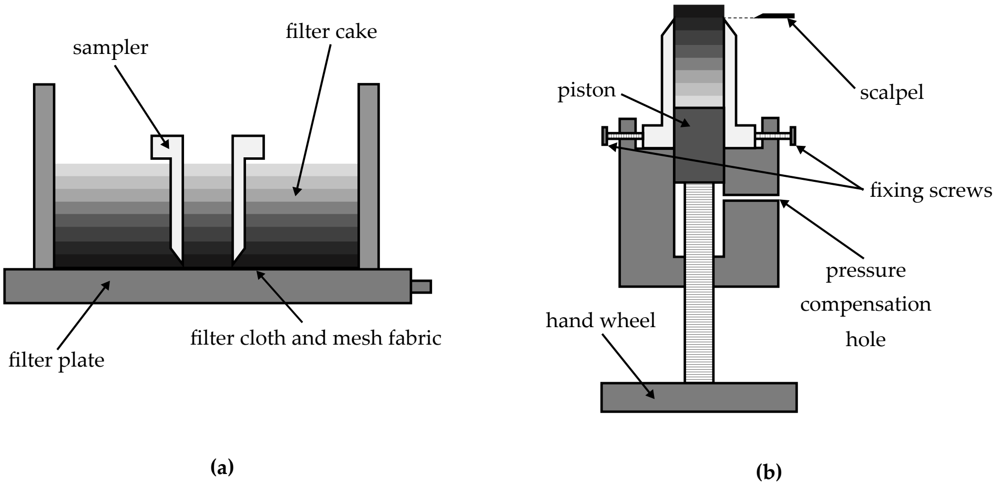


Figure 4. (**a**) Sampling of filter cakes in the laboratory vibration apparatus and (**b**) device for cutting a filter cake sample into several layers.

As the applied gas pressure difference of 80 kPa did not exceed the capillary entry pressure of the filter cakes, the filter cakes were completely saturated in every case. Therefore, determining the residual moisture RM of the filter cake taken with the sampler was a suitable measure for evaluating both the deliquoring and compaction state of the cakes. The cake residual moisture indicates the percentage of liquid mass in the cake m_l relative to the total mass of the wet cake m_{tot} (see Equation (1)). The liquid mass m_l can be replaced by the difference between the total mass of the wet cake m_{tot} and the solid mass of the dry cake m_s. Weighing the filter cake sample before and after drying in an oven at 100 °C for at least 24 h yielded the wet cake mass m_{tot} and the dry cake mass m_s.

$$RM = \frac{m_l}{m_{tot}} \times 100\% = \frac{m_{tot} - m_s}{m_{tot}} \times 100\% \quad (1)$$

To check if vibration application caused particle penetration through the filter cloth, the entire filter cake was removed from the filter cloth after vibration compaction at the highest energy input of 40 Hz and 6000 oscillations. For this purpose, we determined the ratio ϕ_s between the solid mass of the removed filter cake $m_{s,cake}$ relative to the solid mass $m_{s,slurry}$ in the slurry that was initially filled into the filtration unit (see Equation (2)).

$$\phi_s = \frac{m_{s,cake}}{m_{s,slurry}} \times 100\% \quad (2)$$

The determination of the cake's residual moisture RM during squeezing in the CP cell was based on the entire filter cake in the cake formation unit. As full filter cake saturation occurred in the CP cell after cake formation at 80 kPa and during squeezing, like in the vibration apparatus, it was possible to calculate the cake's residual moisture during squeezing with the cake porosity ϵ. The porosity ϵ describes the fraction of the cake pore volume V_{void} in relation to the total cake volume V_{ges}. Replacing the pore volume V_{void} with the difference between the total cake volume V_{ges} and the solid cake volume V_s, the porosity ϵ can be determined using the solid cake mass m_s, the solid density ρ_s, the filter area A, and the cake height $h_{c,load}$ according to Equation (3).

$$\epsilon = \frac{V_{void}}{V_{tot}} = \frac{V_{tot} - V_s}{V_{tot}} = 1 - \frac{\frac{m_s}{\rho_s}}{Ah_{c,load}} \tag{3}$$

Equation (4) shows the calculation of the residual moisture RM using the porosity ϵ, the solid density ρ_s, and the liquid density ρ_l when the cake pores are fully filled with the liquid volume V_l.

$$RM = \frac{m_l}{m_{tot}} \times 100\% = \frac{V_l \rho_l}{V_l \rho_l + V_s \rho_s} \times 100\% = \frac{\epsilon \rho_l}{\epsilon \rho_l + (1-\epsilon)\rho_s} \times 100\% \tag{4}$$

The removal of the entire cake after squeezing in the CP cell was followed by the measurement of the cake height h_c with a laser distance sensor and the determination of the cake residual moisture RM according to Equation (1). The relative cake height increase Δh_c is defined as the difference between the cake height after releasing the compressive pressure h_c and during the pressure load $h_{c,load}$ related to the cake height $h_{c,load}$ during the pressure load in the CP cell (see Equation (5)). This parameter introduced by Wiedemann [39] was used to describe the elastic recovery of the filter cake after squeezing in the CP cell.

$$\Delta h_c = \frac{h_c - h_{c,load}}{h_{c,load}} \tag{5}$$

To examine the deliquoring or compaction state across the cake height after cake formation, squeezing in the CP cell, and vibration compaction, samples were cut for some cases into two layers of equal height following the procedure with the sampler already described. After that, the residual moisture RM of the two layers was analyzed using Equation (1). For the determination of the residual moisture across the cake height, a thicker layer was selected than for the samples used to measure the particle size distribution across the cake height. This is because evaporation effects between sampling and weighing occur that are not negligible if the sample mass and the cake residual moisture are very low.

3. Results and Discussion

3.1. Characterization of Cake Formation for Different Solid Volume Fractions of the Slurry

The filter cake state after formation is the initial state before the subsequent deliquoring step by compaction. Thus, evaluating the impact of particle segregation on the compaction effect by vibration application and by squeezing first required a characterization of the filter cake after formation for the defined solid volume fractions of the slurry. Figure 5 shows the cumulative particle size distributions in the 1 mm thick layers of filter cakes formed from a slurry with a solid volume fraction of 40% (Figure 5a) and 10% (Figure 5b) on the filter plate of the vibration apparatus. As it was not possible to cut layers smaller than 1 mm with the device shown in Figure 4, the uppermost filter cake layer was thicker.

At a solid volume fraction of 40%, the particle size distribution of each filter cake layer was identical (see Figure 5a). This means that the filter cake at this solid volume fraction had a homogeneous structure, where the particles were uniformly distributed across the cake height. In the case of the lower solid volume fraction of 10% shown in Figure 5b, coarser particles were mainly present in the bottom cake layer near the filter medium, while finer particles were located more in the upper cake layers. As the cake height increased, the particle size distribution within the filter cake shifted to the left toward finer particles. This resulted in a heterogeneous filter cake structure due to the particle segregation effect. The selected solid volume fractions are consequently suitable for creating homogeneous and heterogeneous filter cakes for analyzing the impact of the segregation effect on the compaction behavior under oscillatory shear and squeezing. The trends observed in the cumulative particle size distribution $Q_3(x)$ can also be seen in the particle size density distribution $q_3(x)$. This means that it makes no difference for the key statements derived from the particle size analyses if a cumulative or density distribution is used. The cumulative particle size distributions are shown, as the sum parameters $x_{50,3}$, $x_{90,3}$, and $x_{10,3}$, which are often used as characteristic parameters to describe a particle size distribution, can be easily read from it.

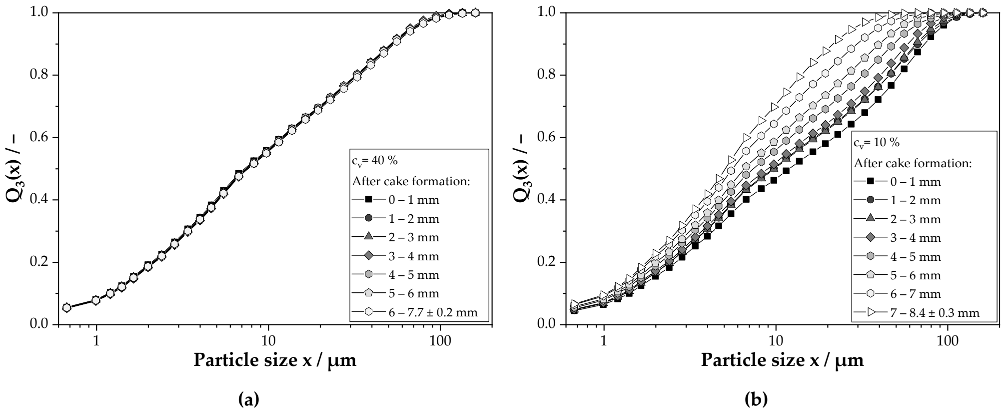


Figure 5. Cumulative particle size distribution in the different layers of a filter cake formed on a filter plate of a vibration apparatus from a slurry with a solid volume fraction of (**a**) 40% and (**b**) 10%. As the coordinate describing the filter cake height starts from the filter medium, the 0–1 mm layer is the lowest cake layer directly on the filter medium.

How homogeneous or segregated cake structures affect filter cake properties such as cake height and residual moisture can be seen in Table 1. The residual moisture values of the homogeneously formed filter cakes at 40% in the bottom layer and in the upper layer are in a similar range due to the even particle size distribution in the cake. There is a slightly smaller residual moisture in the bottom layer, because the solid pressure during cake formation increases in the filtrate's flow direction and reaches its maximum in the cake directly at the filter medium. The result is a porosity gradient across the cake height for compressible filter cakes, which is already described in the literature [37,40]. While the bottom layers close to the filter medium were more compacted, the upper layers had a higher porosity. Due to the complete saturation of the filter cakes investigated in this work, the residual moisture also rose with increasing cake height.

Table 1. Cake height h_c, residual moisture RM in the bottom and top layers after cake formation on the filter plate of the vibration apparatus for different solid volume fractions of the slurry.

c_v /%	h_c /mm	RM /%	
		Bottom	**Top**
40	8.1 ± 0.3	17.9 ± 0.3	18.3 ± 0.4
10	9.0 ± 0.6	17.7 ± 0.3	20.2 ± 0.9

The particle segregation at 10% led to overall higher residual moisture and a greater residual moisture difference between the bottom and upper layers compared to homogeneously formed filter cakes at 40%, which is a familiar consequence of cake formation [11,13,16]. The finer particles in the upper layer form significantly more porous structures with higher residual moisture compared to the bottom layer with coarser particles. As voids between coarse particles are not fully filled by smaller particles as a consequence of particle segregation, the overall structure in segregated filter cakes is less densely packed, and the residual moisture is higher at complete cake saturation than for homogeneously formed filter cakes. This also explains the higher cake height for 10% for a constant solid mass of the slurry.

3.2. Compaction and Dewatering of Homogeneous and Segregated Filter Cakes by Oscillatory Shear

Figure 6 shows the change in residual moisture with the number of oscillations applied to segregated (c_v = 10%) and homogeneously (c_v = 40%) formed filter cakes at frequencies of 17 Hz (Figure 6a) and 40 Hz (Figure 6b).

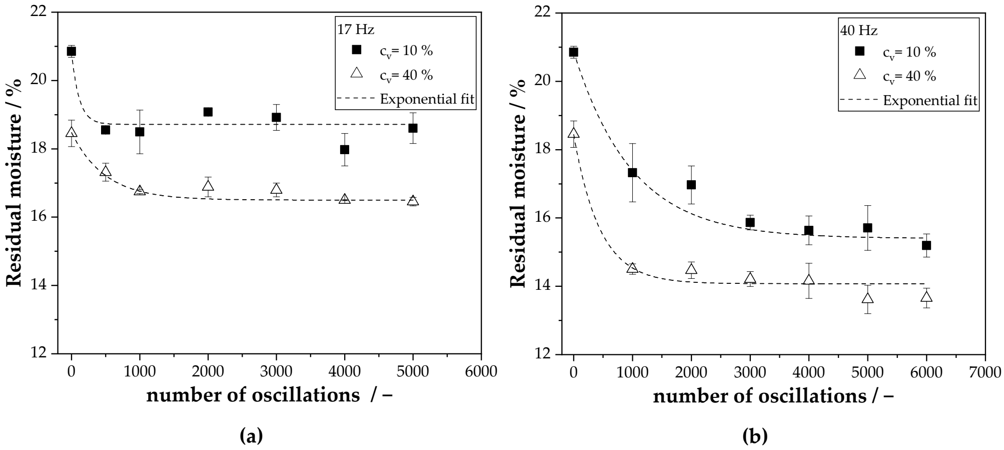


Figure 6. Residual moisture of homogeneous and segregated formed filter cakes after cake formation and vibration compaction at a frequency of (**a**) 17 Hz and (**b**) 40 Hz. The dashed line indicates the approximation of the data using Equation (6).

In general, vibration compaction at both frequencies reduced the residual moisture of the homogeneous and segregated filter cakes with increasing vibration frequency. Increasing the frequency caused a more compacted and drier filter cake for both solid volume fractions. The decrease in residual moisture was exponential in all cases. The cake residual moisture dropped rapidly until the vibration application no longer removed any more liquid from the cake after a few number of oscillations n, and a stationary equilibrium value was reached. The model in Equation (6), already used by Illies et al. [30,31] and Yildiz et al. [36] to describe the exponential kinetics of vibration compaction, provides the minimum achievable residual moisture RM_∞ at a certain frequency in the compaction equilibrium by approximating the experimental data. Other fitting parameters include the consolidation potential B and the compaction rate ϑ.

$$RM(n) = RM_\infty + B \times e^{-\frac{n}{\vartheta}} \tag{6}$$

Table 2 summarizes the adjusted coefficients of determination and the fitting parameters obtained with OriginPro 2019b with the Levenberg–Marquardt algorithm based on the experimental data in Figure 6.

Table 2. Parameters and adjusted coefficients of determination R^2 of the data modeling in Figure 6 with Equation (6). The deviations in the minimum residual moisture RM_∞ and the consolidation potential B are the standard errors of the fit.

Frequency /Hz	c_v /%	RM_∞ /%	B /—	ϑ /—	R^2 /—
17	40	16.5 ± 0.0	2.0 ± 0.4	493	0.8963
	10	18.7 ± 0.1	2.1 ± 0.4	111	0.8352
40	40	14.1 ± 0.2	4.4 ± 0.5	444	0.9274
	10	15.4 ± 0.0	5.5 ± 0.1	990	0.9919

As already mentioned in Section 3.1, the initial state after cake formation and before vibration compaction (zero oscillations) differs depending on the solid volume fraction of the slurry. Due to the homogeneous filter cake structure at 40%, the residual moisture in these cakes was lower than that in segregated filter cakes at 10% before the application of oscillatory shear. After vibration compaction at both 17 and 40 Hz, the minimum achievable residual cake moistures of 16.5 ± 0.0% and 14.1 ± 0.2% for a homogeneously formed cake at 40% were significantly lower than for the segregated filter cakes. Although the use of oscillatory shear at 17 and 40 Hz reduced the residual moisture of segregated cakes to values of 18.7 ± 0.1% and 15.4 ± 0.0%, the compaction or deliquoring effect of the method was significantly weaker compared to that of the homogeneous filter cakes at 40%. As a result, the segregation effect during filter cake formation due to the low solid volume fraction of the slurry limited the success of the subsequent vibration compaction.

In addition to the deliquoring effect, it is crucial for industrial applicability that vibration compaction does not limit the solid mass throughput, and the material retains its particle size distribution after the vibration application. First of all, we needed to check if the particle rearrangement induced by vibration input also triggers particle penetration through the filter medium, diminishing solid mass throughput. For this purpose, the ratios ϕ_s between the solid mass initially applied with the slurry into the filtration unit and the solid mass remaining on the filter medium after cake formation and vibration compaction at the highest energy input studied (6000 oscillations at 40 Hz) are listed in Table 3.

Table 3. Ratio ϕ_s between solid mass on the filter cloth after cake formation and vibration compaction at 40 Hz and 6000 oscillations and solid mass fed with the slurry into the filter plate frame.

c_v /%	ϕ_s /%	
	After Cake Formation	**After Vibration Compaction**
40	99.4 ± 0.1	99.3 ± 0.1
10	99.2 ± 0.0	98.0 ± 0.2

The solid mass remaining on the filter medium after cake formation corresponds almost exactly to the solid mass filled with the slurry in the filtration unit for both solid volume fractions. Particle penetration through the filter medium before particle bridging is thus minimal for both solid volume fractions during cake formation. A higher particle penetration comes from slower particle bridging over the pores of the filter medium at a low solid volume fraction, reflected in the slightly lower ratio ϕ_s for 10% in contrast to 40%. Even after vibration compaction, the solid mass on the filter cloth for the homogeneously formed filter cake is almost identical to the solid mass in the slurry. There is also no change of ϕ_s compared to cake formation. However, there is only a small reduction of ϕ_s after vibration compaction of segregated cakes, although the solid mass loss is still very low. The reason for the higher solid loss for 10% compared to 40% is that the cake adheres more strongly to the filter cloth for the deliquoring state at 10%, making it more difficult to detach the cake completely from the filter medium. Basically, the use of oscillatory shear does not lead to any solid mass loss due to particle penetration through the filter medium.

To verify the particle size distribution after vibration compaction, the particle size of the original particle material and for the the total homogeneous and segregated filter cake samples after cake formation and vibration compaction was analyzed, as shown in Figure 7a,b. For homogeneous filter cakes at 40% and segregated filter cakes at 10%, all particle size distributions lay exactly on top of each other. Overlapping particle size distributions of the original material and the samples after cake formation reveal, like the analysis in ϕ_s, that particle penetration through the filter medium during cake formation was negligible. Since particle size distributions after vibration compaction also matched those after cake formation and the one of the original material, particle crushing or significant penetration of fine particles through the filter cloth due to the vibration input could be ruled out. The particle size distribution, as an important material property, remained the same even after the vibration compaction of homogeneous and segregated filter cakes for the vibration parameters investigated.

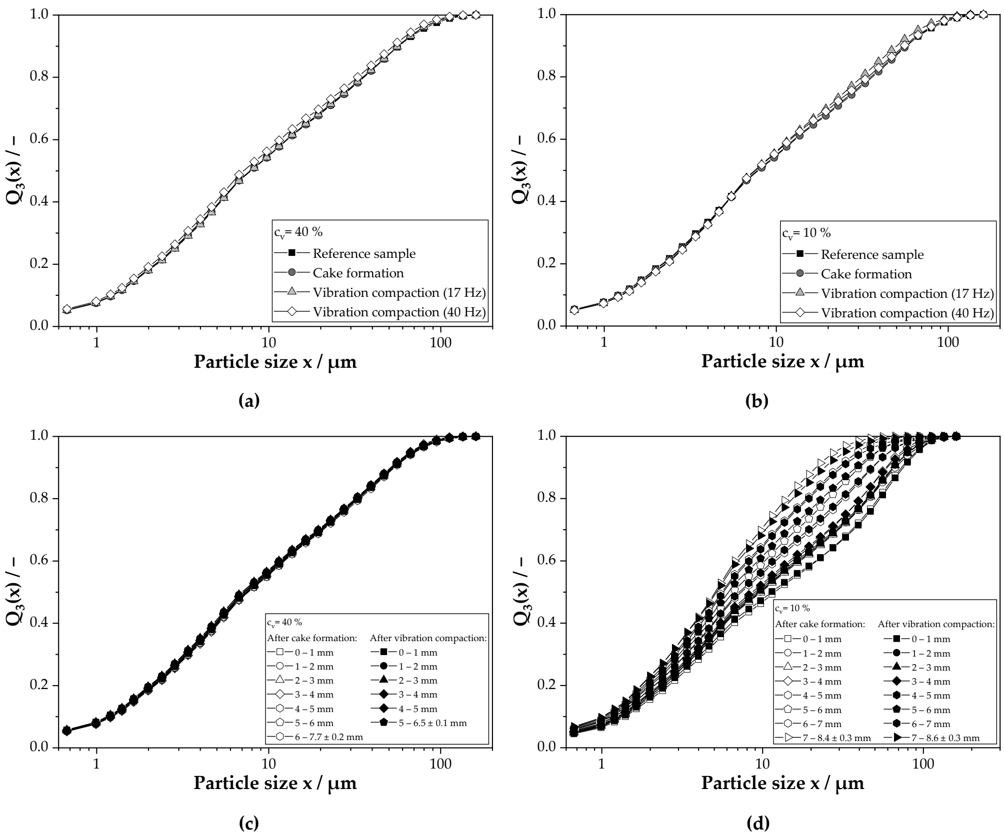


Figure 7. Cumulative particle size distributions of (**a**) homogeneous and (**b**) segregated filter cakes in the full cake height after cake formation and vibration compaction at 17 Hz (1000 oscillations) and 40 Hz (6000 oscillations). The reference samples in (**a**,**b**) represent the particle size distributions of the model material in the original state. Particle size distributions in the different layers of (**c**) homogeneous and (**d**) segregated filter cakes after cake formation and vibration compaction at 40 Hz (6000 oscillations). As the coordinate describing the filter cake height starts from the filter medium, the 0–1 mm layer is the lowest cake layer directly on the filter medium.

The particle size distributions of approx. 1 mm thick cake layers after cake formation and vibration compaction at the highest frequency of 40 Hz in Figure 7c,d were used to characterize the particle rearrangement during vibration compaction. As vibration compaction reduced the cake height of the homogeneously formed filter cake, one layer less than the sample after cake formation was cut out. In a homogeneous filter cake, particle sizes were evenly distributed across the filter cake height after cake formation. Even after vibration compaction, the particle size distributions were identical in all cake layers. Thus, there was a homogeneous particle size distribution across the cake layers cut out after vibration compaction, just like after cake formation. Segregation of the particles, homogeneously distributed across the cake height after cake formation, as a result of the vibration application, did not happen. Since there was no significant height reduction in the segregated filter cake after vibration compaction, the numbers of cake layers cut out after cake formation and vibration input were the same. The shift in the particle size distribution toward finer particle sizes with increasing distance to the filter medium also remained the same after vibration compaction. The particle size distributions in the respective layers were approximately identical after cake formation and vibration compaction. This means that there was no homogenization of the particle size distribution within the cake structure caused by a noticeable movement of finer particles from upper layers into voids between coarser particles in the bottom layers. It could be concluded that particle rearrangement did not occur across a wide range but rather occurred locally at the micrometer scale. This is why the compaction and deliquoring potential of cakes segregatedby vibration compaction are significantly smaller compared to those of homogeneous cakes.

Table 4 displays the achievable residual moisture in the bottom and upper layers of the homogeneous and segregated filter cakes after vibration compaction to the equilibrium state. For both solid volume fractions, a decrease in residual moisture in both the bottom and upper cake layers with oscillatory shear compared to the original state after cake formation (see Table 1) was evident. In both cases, increasing the frequency improved the mechanical deliquoring by compaction in the bottom and upper cake layers. This indicates that the mechanical power from the oscillatory plate on the filter cake surface reached not only the upper cake area near the plate but also the bottom cake layer. For homogeneous filter cakes at 40%, the deliquoring states in the bottom and top layers were similar after vibration compaction and cake formation. In the case of segregated filter cakes at 10%, the residual moisture difference between the bottom and top layers, which was already present after cake formation, also existed after vibration compaction. The upper cake layer continued to have a considerably higher residual moisture than the bottom cake layer. As vibration application did not induce a homogeneous particle dispersion between finer particles in the upper layer and coarser particles in the bottom layer (see Figure 7), fine particles did not fill all voids between coarser particles. Hence, the same compaction state as for a homogeneously formed filter cake could not be achieved.

Table 4. Residual moisture *RM* in the bottom and top layers of homogeneous and segregated filter cakes after vibration compaction.

Frequency/Hz	c_v /%	*RM* /%	
		Bottom	Top
17	40	15.8 ± 0.2	16.3 ± 0.2
	10	16.4 ± 0.4	19.7 ± 0.3
40	40	13.6 ± 0.8	13.8 ± 1.0
	10	13.5 ± 0.1	15.6 ± 0.3

3.3. Compaction and Dewatering of Homogeneous and Segregated Filter Cakes by Squeezing

Figure 8 compares the minimum achievable residual moisture of homogeneous and segregated filter cakes by squeezing in the CP-cell during and after the pressure load (black and white data points) and after vibration input (horizontal lines).

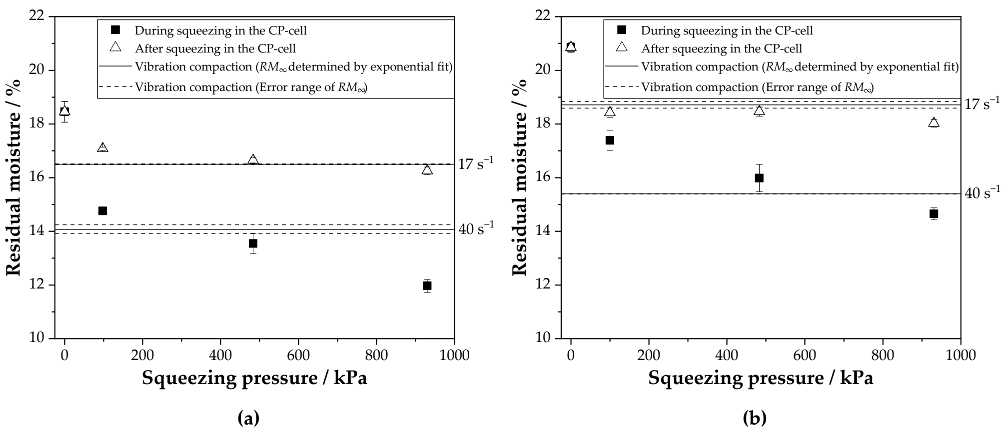


Figure 8. Minimum residual moisture of (**a**) homogeneous and (**b**) segregated filter cakes during and after squeezing at different pressures in the CP-cell and after vibration compaction. The values at a squeezing pressure of 0 kPa are the reference cake conditions after cake formation.

Squeezing the filter cakes decreases residual moisture considerably, although deliquoring by squeezing is more effective for homogeneous filter cakes than for segregated filter cakes. The compaction and deliquoring effect grows with increasing pressure. Regarding the states of homogeneous filter cakes during load by the compressive pressure, only vibration compaction at the highest frequency of 40 Hz can keep up with squeezing up to 480 kPa. Vibration compaction at 40 Hz actually achieves a slightly smaller residual moisture for a segregated filter cake in comparison to squeezing at 480 kPa. In both cases, increasing the squeezing pressure to 1000 kPa removes more water from the filter cake than vibration compaction up to 40 Hz. In terms of the deliquoring effect, compaction by squeezing has an advantage over vibration compaction.

But, in industrial reality, squeezing compressible filter cakes at such high pressures demands a special design for the filtration apparatus, which is associated with higher costs, so that the apparatus components can cope with the high load caused by the squeezing pressures. However, implementing vibration compaction on an existing continuous filter apparatus with a simple design is feasible [33]. The filter cake is relieved again after squeezing during industrial operation, enabling the possible elastic recovery of the compressible filter cake to occur. Moreover, we can only consider the achievable cake deliquoring and compaction states of vibration compaction after the application. For these reasons, Figure 8 illustrates the residual cake moisture after compaction by squeezing to allow a comparison of the two processes under the same conditions. It is noticeable that the residual moisture of the homogeneous and segregated filter cakes were distinctly higher after pressure relief than during pressure loading. The explanation for this is that the elastic recovery behavior of the filter cake causes the cake to reabsorb the previously squeezed liquid that is stored in the filter cloth, in the support fabric, in the perforated plate, or in the filtrate drain bottom as residues. This behavior impairs the deliquoring efficiency of squeezing. Mazzi and Krammerer [41] already observed this behavior for coal filter cakes under simultaneous compression and steady shear as in belt filter presses. Wiedemann [39] and Alles [37] observed elastic recovery behavior after compressing filter cakes of mineral, fibrous, deformable, and finely agglomerated particles. The relative cake height increase in Table 5 characterizes the elastic recovery behavior of the filter cakes after squeezing.

Table 5. Relative cake height increase Δh_c of homogeneous and segregated filter cakes after squeezing at different pressures in the CP cell.

Pressure/kPa	Δh_c **/%**	
	c_v **= 40%**	c_v **= 10%**
100	11.9 ± 1.2	8.6 ± 0.4
480	11.9 ± 0.4	10.6 ± 1.8
930	15.9 ± 1.0	13.1 ± 1.5

The relative cake height increase of the homogeneous filter cakes rose with the pressure from 11.9% for 100 kPa to 15.9% at 930 kPa, while the parameter increased from 8.6% at 100 kPa to 13.1% at 930 kPa for the segregated filter cakes. The lower compaction state during pressure load explains the overall smaller elastic recovery of the segregated filter cakes compared to that of the homogeneous filter cakes. Comparing the residual moisture after both compaction methods, vibration compaction showed advantages in terms of the deliquoring and compaction effect for the two differently formed filter cakes. At 17 Hz, the residual moisture levels were still roughly in the range of compaction by squeezing up to 930 kPa. Nevertheless, vibration application compacted the cakes at 40 Hz to a greater extent than squeezing. To prevent cake rewetting due to elastic recovery after pressure relief, it is essential to completely remove the liquid contained in the filter cloth and support fabric as well as the residual liquid in the filtrate drain during pressure load. The CP cell was completely sealed during pressure load, preventing air from flowing through the filter cloth, the support fabric, and the perforated plate due to the applied vacuum to replace and remove the liquid stored there. In industrial applications, a bypass airflow past the filter cake through the filter cloth, e.g., on drum or belt filters, can be implemented in principle. But, this leads to higher operating costs resulting from increased gas consumption of the vacuum pump and to a reduction in the pressure difference in the worst case.

Table 6 lists the achievable residual moisture in the bottom and top layers of the filter cakes after squeezing at 100 and 930 kPa in the CP cell. Squeezing homogeneous filter cakes at both pressures compacted both the bottom and top layers. The residual moisture values in the top and bottom layers after squeezing homogeneous filter cakes were similar, as observed after vibration compaction. In the case of the segregated filter cakes, a residual moisture difference between the bottom and top cake layers, which already existed after cake formation, could still be seen as after vibration compaction. The difference to vibration compaction is that the residual moisture of the bottom filter cake layer after squeezing up to 930 kPa does not change compared to the state after cake formation. Compaction by squeezing only acted on the upper layer with the finer particles, while vibration compaction achieved significant compaction or mechanical deliquoring in both layers. The deeper impact of applying oscillatory shear ensures more effective mechanical deliquoring of segregated filter cakes in contrast to squeezing.

Table 6. Residual moisture *RM* in the bottom and top layers of homogeneous and segregated filter cakes after squeezing in the CP cell.

Pressure/kPa	c_v /%	*RM* /%	
		Bottom	Top
100	40	16.3 ± 0.7	16.7 ± 0.5
	10	17.9 ± 0.6	19.1 ± 0.3
930	40	15.3 ± 0.1	15.7 ± 0.2
	10	17.9 ± 0.1	18.6 ± 0.3

4. Conclusions

Particle segregation, occurring at low solid volume fractions of the slurry, lowers the cake deliquoring efficiency of vibration compaction. The results of the particle size analyses of approx. 1 mm thick layers cut from filter cakes showed that vibration input does not cause wide-ranging particle rearrangement within the cake. Fine particles in the upper layers do not migrate completely into the voids of coarser particles in the bottom layers, which means that the same compaction condition is not achieved as for a homogeneous filter cake. Therefore, a high solid volume fraction of the slurry is desirable for a homogeneous filter cake structure to achieve high deliquoring efficiency by vibration compaction. Furthermore, particle crushing and penetration through the filter cloth as a result of vibration application were ruled out.

The deliquoring results of vibration compaction were significantly lower than those of squeezing, since the elastic recovery behavior of the filter cakes after the pressure relief during squeezing reabsorbs the liquid already squeezed from the filter cloth, the support structure, and the filtrate drain bottom. A bypass airflow through the filter cloth and the support structure can displace the liquid stored there by a vacuum applied during squeezing to prevent rewetting and to achieve increased deliquoring compared with oscillatory shear. Unfortunately, the higher gas throughput increases operating costs. Higher investment costs for the apparatus design, arising from the heavy pressure load on the apparatus components due to a press module, must also be taken into account for the squeezing method. Overall, vibration compaction, which can be implemented on an existing belt filter [33], is a real alternative for the mechanical deliquoring of mineral filter cakes.

To make particle rearrangement visible and further clarify the compaction mechanism, it would be interesting to use imaging methods with a higher resolution in the future. A higher-resolution study of the vibration compaction effect within the filter cake, rather than just considering the bottom and top halves of the cake as in this study, would also be informative.

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Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

The following abbreviations are used in this manuscript:

CP cell	compression-permeability cell

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