

Application of a Particle Extraction Process at the Interface of Two Liquids in a Drop Column—Consideration of the Process Behavior and Kinetic Approach

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Abstract

The focus of this research is a new type of particle extraction process for the transfer of magnetite nanoparticles from an aqueous to an immiscible organic phase, directly through the liquid-liquid phase boundary in a drop column. The particle extraction process comprises several advantages such as a minimum amount of stabilizing surfactant, no exposure of the particles to a gas atmosphere and with it the avoidance of sintering by capillary forces and a high particle concentration in the receiving phase as well. The study presents experimental results of the characterization of the process environment and the transfer behavior in a drop column. The solution of surfactant in the continuous phase has been investigated during a particle-free phase transfer experiment including the measurements of the total organic carbon (TOC) content and analysis of the size of the stabilized droplets using the laser diffraction spectroscopy. The determination of the transfer fluxes, the mass flows as well as the yield of transferred magnetite by ICP-OES measurements provide information on the impact of interaction of the elementary processes at the phase boundary. Furthermore, the transfer kinetics of the process is described and compared with calculated theoretical values resulting from a kinetic approach.

Keywords

Magnetite, Nanoparticles, Particle Extraction, Drop Column, Ricinoleic Acid, Oleic Acid

1. Introduction

Because of their special magnetic properties, magnetite nanoparticles have a great potential for many technological applications. Therefore, they are very interesting for a broad range of research areas, for example as magnetic fluids for low friction dynamic gasket systems as well as for the construction of vibration dampers and tweeters [1] [2]. A particular research focus represents the application of magnetite nanoparticles as advanced functional materials for surface coatings and particle composite materials [3] [4].

Possible areas of application for this can be found in the field of reaction engineering [5], in the form of magnetically separable catalyst material as well as in the biomedical sector [6]-[8]. Especially in combination with polymers, stabilized magnetite nanoparticles are required in an organic phase [9]-[12]. However, the nanoparticles are synthesized mainly in an aqueous phase and have to be placed in an organic phase through appropriate procedures. Due to their increased surface area/volume ratio, nanoparticles are susceptible to oxidation and have a great tendency to agglomerate, which may cause a loss of their special magnetic properties. As a consequence, conventional transfer strategies, based on filtration with subsequent drying and redispersion steps, can only be applied conditionally [13]. Therefore, the development of an efficient process to transfer the magnetite nanoparticles from the aqueous to an immiscible organic phase is of great interest.

Emphasis should be put on producing stable colloidal and functionalized particles continuously with a minimum use of surfactants in the liquid organic medium. To demonstrate the continuous phase transfer process via a particle extraction, we design a concept of a miniplant using a drop column as the chosen transfer device. In the literature, the bubble columns with different internals are thoroughly investigated regarding to mass transfer, flow patterns, bubble shapes and hydrodynamics, for example with applications in metallurgical, chemical, bio- and petrochemical process industries [14]-[17]. In our study, a column is used in the simplest form without internals. An organic liquid as disperse phase is injected as drops through a distributor into a magnetite nanoparticle suspension, which represents the continuous aqueous phase. Already, in 1968 Lai and Fuerstenau [18] have carried out a liquid-liquid extraction of ultrafine particles to separate a mixture of alumina, water and oil, however they use for their experiments a separatory funnel to put on top of each other the two phases.

In this study, the particle extraction process is investigated and the results are presented with the drop column in a partial recirculation operation. This means that the aqueous phase is stationary and that the organic phase is in recirculation, as shown in **Figure 1**. This is a first step to the future operation with a closed circuit of both phases.

The process mechanism of the particle transfer in the drop column can be divided into three fundamental steps. At first, the nanoparticles agglomerate partly due to the instable conditions in the aqueous phase as already mentioned in [19]. In the drop column, after injecting the organic phase, no sedimentation takes place due to the stirring effect of the rising liquid drops. Particle drop collision occurs. At the interface, the surfactants from the organic phase interact with the hydrophilic particle surface. The surfactant molecules adsorb and become chemically grafted [20] [21]. This leads to a hydrophobization and functionalization of the particles, which allows a phase transition of the magnetite.

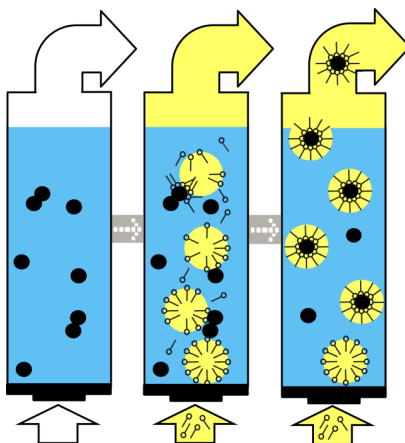


Figure 1. Scheme of the fundamental steps in the liquid-liquid particle extraction process mechanism in the drop column as transfer device.

Depending on the surfactant used, stable organic colloids are formed by an excellent deagglomeration of the particles and a physicochemical dispersion due to the strong repulsive potential of the adsorbed surfactant molecules [12].

The aims for the phase transfer process in the drop column are a high efficiency, which means a high yield of transferred magnetite in the organic phase, low transfer times or rather process times and a stable product. Furthermore, a stable process in the drop column is necessary. It means a stable drop formation and upward movement with a sufficient coalescence rate, whereby a clearing off of the column is essential for the setup chosen.

2. Materials and Methods

The synthesis of the magnetite nanoparticles with a crystallite size of about 15 nm [21] by a wet-chemical coprecipitation reaction is carried out at 70°C under atmospheric conditions as described by Machunsky *et al.* [13]. Therefore we applied the precursors iron (II) sulphate heptahydrate and iron(III)chloride hexahydrate purchased from Carl Roth Germany as well as the precipitant ammonium hydroxide solution with an ammonia content of 26% from Sigma.

For the phase transfer experiments in the drop column the pH-conditioning of the aqueous phase is necessary; otherwise emulsion formation in the drop column occurs. The aqueous suspension is conditioned under ambient air by repeated washing with distilled water to a pH value of 4 - 5. For the experiments with oleic acid (OA) the original salt concentration of 39.4 g/l and with ricinoleic acid (RA) a quarter of the original salt concentration is utilized. Subsequently, for both surfactants used a process-pH value of 8.10 ± 0.05 is adjusted.

The research of the liquid-liquid particle extraction process in the drop column is effected by the choice of the surfactants, ricinoleic acid and oleic acid respectively. Both are unsaturated fatty acids (FA) at the 9th C-atom with a carbon chain length of 18 C-atoms. RA has an additional hydroxyl-group at the C-atom C12. The surfactants are of technical grade with 90% purity and purchased from Sigma.

The organic phase as the disperse phase is consisting of the solvent iso-octane provided by Carl Roth Germany with 99.5% purity as well as the mass fraction of surfactant x_{surf} and a specific amount of surfactant per magnetite $X_{\text{S/M}}$. The study presents the results with the parameters $x_{\text{surf}} = 1.4$ mass-% and $X_{\text{S/M}} = 0.2$ g/g.

All chemicals are used as received.

The magnetite mass concentration is determined with ICP-OES analyses (inductively coupled plasma optical emission spectroscopy) of Fe with the ICP spectrometer iCAP 6300 from Thermo Fischer Scientific. For the measurements, 5 emission lines of Fe with different wave length ($\text{Fe}_{238.2 \text{ nm}}$, $\text{Fe}_{240.4 \text{ nm}}$, $\text{Fe}_{259.9 \text{ nm}}$, $\text{Fe}_{274.6 \text{ nm}}$, $\text{Fe}_{274.9 \text{ nm}}$) are used, which cover the whole mass concentration range. For this, the samples were chemically digested with concentrated hydrochloric acid delivered from Carl Roth Germany. For each emission line a triple determination was performed, resulting with a relative standard deviation of <1%.

The total organic carbon (TOC) content measured with the device from analytikjena multi N/C 2100s is determined by using the difference method. This means the total carbon (TC) content is analyzed. After degassing the inorganic carbon (IC) content is measured and subsequently the TOC content can be calculated. If the IC content is negligible the TC-method is used, in which the TC content is measured directly, and this value corresponds with the TOC content.

For the analysis of the size distribution of stabilized droplets by laser diffraction spectroscopy the spectrometer HELOS, manufactured by Sympatec, is used. The measurement range for this device is between 0.1 - 875 μm .

Experimental Setup

As mentioned the drop column as transfer device is used in a partial recirculation operation, that means the aqueous phase is stationary and the organic phase is in recirculation, as seen in **Figure 2**. The drop column has a length of 700 mm with an internal diameter of 25 mm. At the bottom of the column the distributor (sparger) as dispersing system is a single metal capillary with an inside diameter of 3.2 mm centrally mounted in a perforated plate. The mixing of the organic phase in the receiver tank is ensured by a mechanical agitator.

Limited by the dimensions of the transfer device a volume of 300 ml of the magnetite suspension, which corresponds to a mass of 6 g magnetite nanoparticles, is filled into the column as continuous phase. The organic phase, which acts as disperse phase is pumped from the receiver tank through the metal capillary into the drop

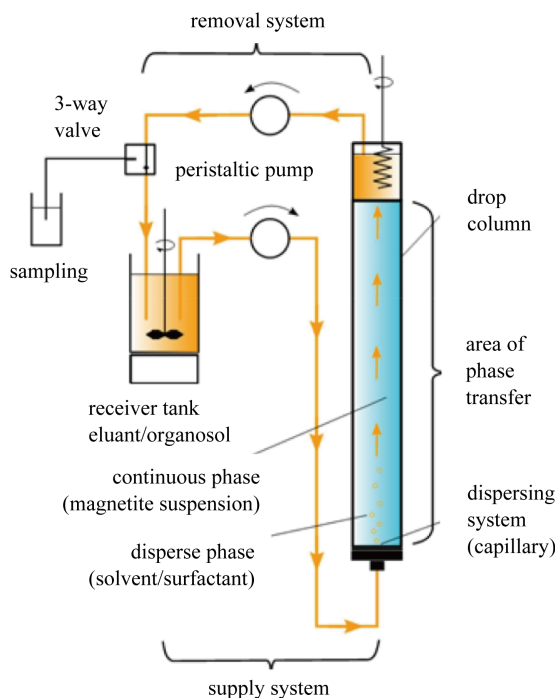


Figure 2. Experimental setup of the miniplant in a partial recirculation operation: aqueous phase stationary and organic phase in recirculation.

column by a supply system consisting of a peristaltic pump (Ismatec Reglo Analog) and a Tygon-tube with an inside diameter of 3.2 mm. Directly above the opening of the metal capillary, whereby dead zones can be formed aside, the organic phase immediately disintegrates in differently sized drops. These rise through the column in the aqueous phase, due to their lower density. The drops transport the extracted magnetite and thus the nanoparticles are concentrated within the coalesced organic phase. The presence of the solvent drops within the column leads to an expansion of the liquid level, the so-called hold-up.

The organic phase containing the extracted magnetite particles is collected at the top of the liquid and pumped back to the receiver tank through the removal system. Due to the fact that surfactants are used, the rising drops can form a so called dispersion ribbon at the surface of the continuous phase. This is a layer consisting of droplets and only partial hydrophobized particles, where the coalescence is hindered [22]. The removal system comprises a peristaltic pump and Tygon-tubes with the same properties as in the supply system as well as an automated sampling system with a pneumatic 3-way valve, manufactured by Swagelok, which is controllable via measurement software.

Due to the hydrodynamic dead time the first measuring point can be taken after 85 seconds, because then organosol is existent in the removal system. The average residence time in the column is about 51 seconds and the volume flow rate is 29 ml/min. The sample volume amount is 2.9 ml and is added at the same time manually as pure organic phase in the receiver tank, hereby keeping the volume of the disperse phase constant during the recirculation. However, after each removed sample volume the amount and mass concentration of magnetite nanoparticles in the system is reduced, which is included in the calculation of $\beta_{magn,rt}(t)$ based on the validation of the system.

For the evaluation of the corrected mass concentration of transferred magnetite nanoparticles in the receiver tank $\beta_{magn,rt}(t)$ a verification of the system is applied taking into consideration the input and output to the receiver tank as well as the residence time distribution in Equation (1):

$$\beta_{magn,rt}(t) = \beta_{magn,rt,0} \times e^{-\frac{\dot{V}_{disp}}{V_{rt}}t} + \int_{t'=0}^{t'=t} \frac{\dot{V}_{disp}}{V_{rt}} \beta_{magn,in}(t) \times e^{-\frac{\dot{V}_{disp}}{V_{rt}}(t-t')} dt \quad (1)$$

where $\beta_{magn,rt,0}$ is the initial mass concentration of the system, V_{rt} is the volume in the receiver tank, \dot{V}_{disp} is

the volume flow of the disperse phase and t' is the auxiliary variable, which describes the time of the entry.

By linear interpolation and numeric integration, the Equation (1) can be solved. This results in the following equation:

$$\beta_{magn,rt}(t) = \left(\beta_{magn,rt,0} + \frac{a_i \times V_{rt}}{\dot{V}_{disp}} - b_i \right) \times e^{-\frac{V_{rt}}{\dot{V}_{disp}} t} + a_i t + b_i - \frac{a_i \times V_{rt}}{\dot{V}_{disp}} \quad (2)$$

where a_i as well as b_i are formed by linear interpolation between the removed samples.

To characterize the process environment we have investigated the procedures in the drop column by the method of a particle-free operation, whereby the changes in the column can be observed. At defined time points the pumps were switched off and the sampling is effected from the continuous phase. In dependence from surfactants used the different mechanisms of drop formation of the disperse phase in the column is demonstrated in **Figure 3**. With ricinoleic acid as surfactant (**Figure 3(a)**) the disperse phase disintegrates in a multitude of very small droplets and turbidity appears in the column at once, which spreads in the whole column within a few minutes. In contrast, with oleic acid (**Figure 3(b)**) as surfactant a single periodic drop formation [23] occurs resulting in larger and significantly less drops without turbidity.

3. Results and Discussion

As mentioned above the both surfactants used have a different process behavior with varying consequences to the particle extraction process. The turbidity in the column with ricinoleic acid as surfactant does not influence the process stability of the phase transfer, because the disperse phase can be performed in recirculation without hindrance. The reason for the formation of small emulsion droplets is due to the mass transport of ricinoleic acid through the phase boundary. This is possible, because ricinoleic acid is soluble in the organic as well as in the salts and ammonia containing aqueous phase owing to the additional hydroxyl group. This effect of the spontaneous formation of an emulsion has been described relating to a pH value dependency of Stackelberg, 1949 [24]. In our case an imbalance prevails in the column at the interface of the rising drops, since the ricinoleic acid is dissolved in the organic phase, only at beginning of the process. The system aspires an equalization of concentration by the transport of ricinoleic acid through the phase boundary into the aqueous phase. The resulting convection currents cause deformations and lead to the formation of very small droplets. **Figure 4** outlines these stabilized iso-octane droplets with a median value of 5 - 10 μm measured by laser diffraction spectroscopy. They can only be formed if ammonium ricinoleates (carboxylates) exist at the interface by dissolving of ricinoleic acid in the aqueous phase, which definitely reduces the interfacial tension [25]. Furthermore, the smallest median value of the stabilized iso-octane droplets is reached after 20 minutes of defined time points. Due to the low buoyant force of these droplets, the turbidity is appearing in the column.

To prove the solution of surfactants from disperse into continuous phase the TOC content in the aqueous phase is determined and plotted as a function of the time, as shown in **Figure 5**. By additional centrifugation of the aqueous phase, a potential carbon signal of emulsified solvent can be excluded (light gray columns as short-time trial and dark gray columns as long-time trial). By using ricinoleic acid (**Figure 5**, top) as surfactant, a significant increase of the TOC content can be observed. After 5 minutes process time the aqueous phase exhibits an amount of dissolved ricinoleic acid in the range of 1.6 - 2.4 g/l independent of the time limit of experi-

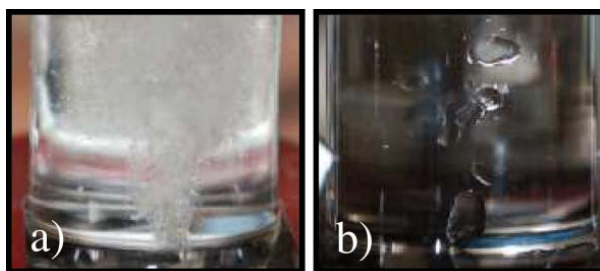


Figure 3. Drop formation of the disperse phase in the column directly above the distributor with ricinoleic acid (a) and oleic acid (b) as surfactant at beginning of the process.

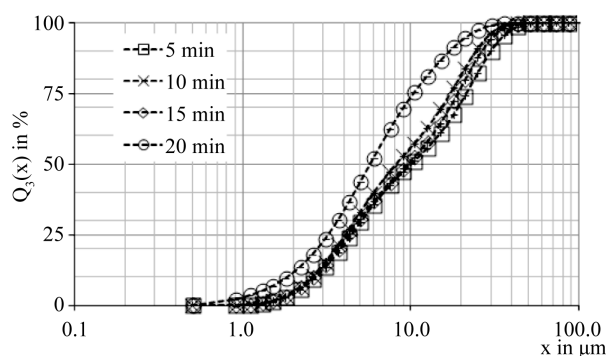


Figure 4. Size distribution of the stabilized iso-octane droplets in the aqueous phase with ricinoleic acid as surfactant using laser diffraction spectroscopy.

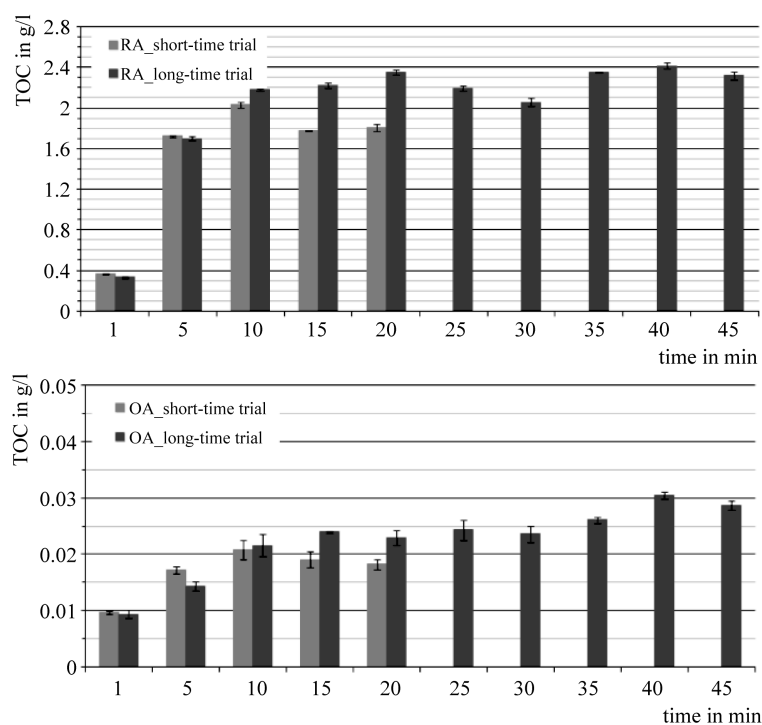


Figure 5. Total organic carbon (TOC) content at defined time points of sampling in the aqueous phase by centrifugation of the partly emulsified aqueous phase (light gray columns as short-time trials and dark gray columns as long-time trials) with ricinoleic acid (top) and oleic acid (bottom) as a surfactant.

mental procedure. Whereas oleic acid (**Figure 5**, bottom) is used, the poor ability of dissolving within the aqueous phase is expressed considerably.

So the TOC values of oleic acid actually dissolved in the aqueous phase are nearly in the range of 0.01–0.03 g/l. Comparing the average values of the mentioned ranges (2 g/l for ricinoleic acid and 0.02 g/l for oleic acid), it can be recognized that in contrast to oleic acid approximately the 100-fold amount of ricinoleic acid is dissolved in the aqueous phase irrespective of time.

Furthermore, experiments are performed with a pH-indicator bromothymol blue in the continuous phase, as demonstrated in **Figure 6(a)**, which has a transition point at pH 7.6 (color change from blue to green). For either surfactant an initial process-pH value of 8.1 is adjusted, similar to the conditioned aqueous phase for the phase transfer of magnetite. Only with ricinoleic acid in the disperse phase a change from blue to green is observed in

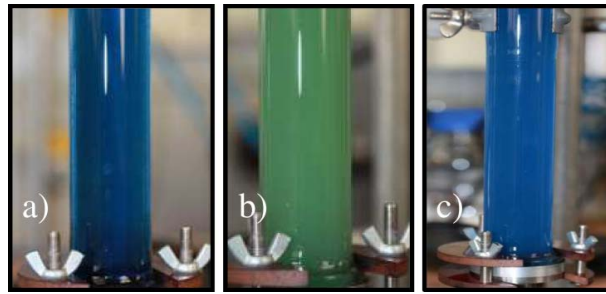


Figure 6. Characterization of the pH value changes in the continuous phase using the indicator bromothymol blue (a) after recirculation of the disperse phase with ricinoleic acid (b) and oleic acid (c) as a surfactant.

the continuous phase (**Figure 6(b)**). This indicates that the pH value has decreased below 7.6. The reduction in the concentration of ammonium ions is initiated by the formation of ammonium ricinoleates at the liquid-liquid interface due to the dissolving of ricinoleic acid in the aqueous phase. After the recirculation of the disperse phase with oleic acid as surfactant the color of the continuous phase has changed to light blue (**Figure 6(c)**), because significantly less of oleic acid is dissolved in the aqueous phase. As a result the formation of ammonium oleates at the interface is lower, which only leads to a slight change of pH in the aqueous phase.

This finding implies that more than one process is relevant at the phase boundary.

For the characterization of the transfer behavior the yield of transferred magnetite particles $\phi_{\text{magn,trans}}$ is plotted against the process time in **Figure 7**. This parameter is determined, from the time dependent corrected magnetite mass concentration in the receiver tank $\beta_{\text{magn,rt}}(t)$ based on the validation of the system in Equation (1) relating to the theoretical applied magnetite mass concentration for the phase transfer $\beta_{\text{magn,PT,theor}}$ in the following equation:

$$\phi_{\text{magn,trans}} = \frac{\beta_{\text{magn,rt}}(t)}{\beta_{\text{magn,PT,theor}}} \quad (3)$$

Based on the short-time trial with the process time limit of about 10 minutes it can be observed excellently, that the phase transfer with ricinoleic acid as a surfactant takes place rapidly. The increase of the yield of transferred magnetite is nearly twice the amount compared to oleic acid. This is consistent with the significant increase in TOC content, because by dissolving of ricinoleic acid in the aqueous phase the drops disintegrate more easily and thus an additional phase boundary can be formed for the phase transfer process. Due to the lower solution of oleic acid in the aqueous phase the available phase interface is smaller, which leads to longer process times for the phase transfer of magnetite. This also can be observed during the long-time trials with both surfactants. With ricinoleic acid it can be demonstrated, that already after 15 minutes the yield of transferred magnetite amounts to approximately 90% and it is kept constant about over time, up to 165 minutes. However, with oleic acid the transfer is completed after nearly 270 minutes and the maximum with a yield of transferred magnetite of about 86%. Furthermore, the clearing off of the column with ricinoleic acid as surfactant is obtained after about 6 minutes, whereas with oleic acid it can be achieved only after 160 minutes.

For the characterization of transfer kinetics, the mass flow of transferred magnetite particles $\dot{m}_{\text{magn,trans}}$ is plotted against the process time in **Figure 8**. This parameter is determined, by using the constant volume flow of the disperse phase \dot{V}_{disp} and the difference in mass concentration of transferred magnetite between output and input of the disperse phase $\Delta\beta_{\text{magn,out,in}}(t)$ in Equation (4).

$$\dot{m}_{\text{magn,trans}} = \dot{V}_{\text{disp}} \times \Delta\beta_{\text{magn,out,in}}(t) \quad (4)$$

The plot of the mass flow with ricinoleic acid as a surfactant is conspicuously distinguished by the increase from the first measuring point at 85 seconds till the maximum of the mass flow at a process time between 4 - 5 minutes. During this initial phase the ricinoleic acid concentration in the aqueous phase still rises, which limits the mass flux of magnetite. Subsequently, the mass flow is described with a kinetic of 1st order as it is also identified with oleic acid as surfactant, what needs to be proved.

In the present case the product of functionalized magnetite particles (B) are formed from the precursor of pure

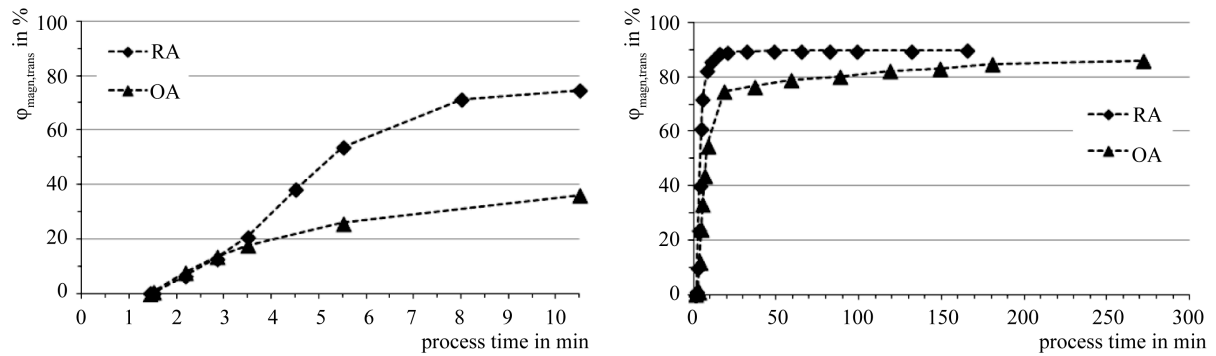


Figure 7. Yield of transferred magnetite in dependency of the process time for ricinoleic acid and oleic acid as a surfactant during short-time and long-time trials.

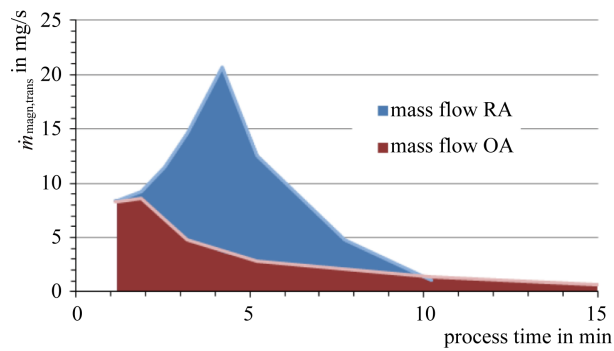
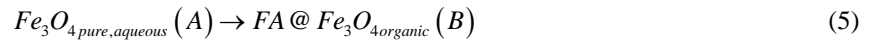


Figure 8. Demonstration of the time dependent mass flow of transferred magnetite particles dependent on the surfactant used.

magnetite nanoparticles (A) in Equation (5).



It is supposed that the already transferred magnetite concentration in the organic phase does not influence the transfer process. With this assumption the drop column can be seen as conventional tank reactor [26], in which the concentration of magnetite is reduced by the transfer reaction.

Thereby, the differential temporary law of the reaction can be established in Equation (6). This indicates, that the temporal change of mass concentration $d\beta_A/dt$ is proportional to the currently existing mass concentration β_A .

$$\frac{d\beta_A}{dt} = -k\beta_A \quad (6)$$

where k is the rate constant of reaction (1/s) of the 1st order and the product $k\beta_A$ is the rate of consumption of A. Furthermore, the half-life $t_{1/2}$ for the reaction of both surfactants can be determined from the experimental data. The half-life is the time, at which half of the precursor is dissipated. By means of Figure 9 it can be distinguished, that this corresponds to the point of intersection, between the normed mass concentration of the precursor β'_A and the product β'_B . Due to the comparability a dimensionless representation of the mass concentration is chosen by the scaling in Equation (7).

$$\beta' = \frac{\beta_{\text{magn, ICP}}}{\beta_{\text{max}}} \quad (7)$$

The relative change in the mass concentration $d\beta_A/\beta_A$ in Equation (8) is proportional to the temporal change dt . It can be calculated from the experimental data by the ICP-OES measurements and is demonstrated in Figure 10 (plots using filled symbols).

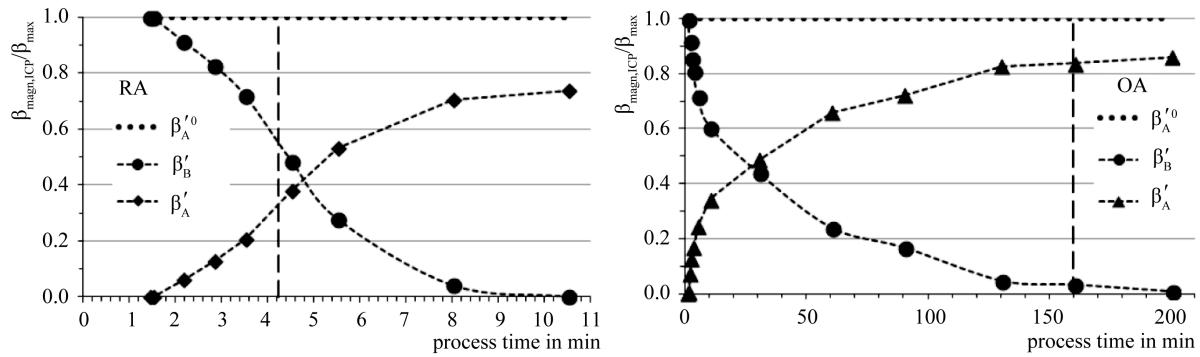


Figure 9. Dependency of the half-life of surfactant used presented by the normed mass concentration plots—with ricinoleic acid (left) the half-life is 4.78 minutes, whereas the half-life with oleic acid (right) is 27.2 minutes. The dashed line indicates the clearing off of the column.

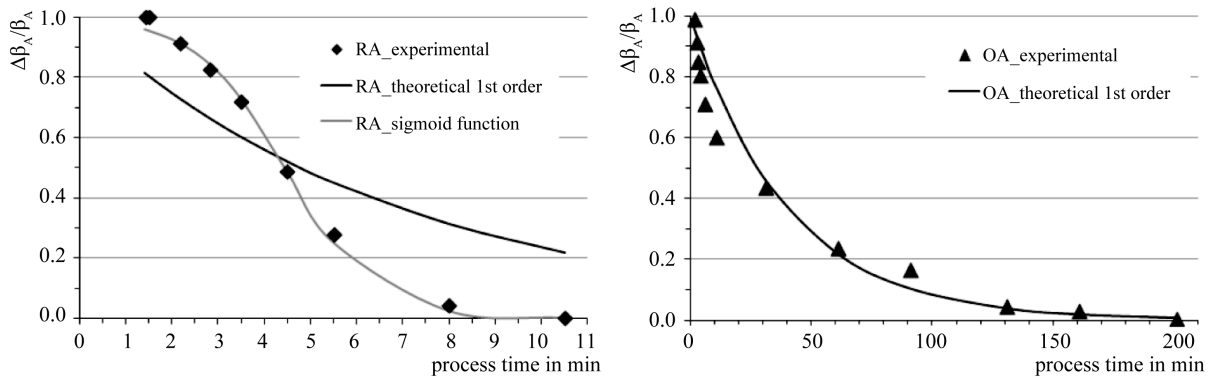


Figure 10. Demonstration of the experimental as well as theoretical calculated relative change in the mass concentration in dependency of the process time with ricinoleic acid (left) and oleic acid (right) as a surfactant.

$$\frac{d\beta_A}{\beta_A} = -kdt \quad (8)$$

By integration from $t = 0$ till end time t as well as from initial mass concentration β_A^0 till $\beta_A(t)$ and additional by the use of logarithmic, the integral temporary law is achieved in the following equation:

$$\beta_A = \beta_A^0 \times e^{-kt} \quad (9)$$

resulting in the determination of the theoretical mass concentration β_A and using the Equation (10) for the calculation of k for both surfactants derived from Equation (9)

$$k = \frac{\ln 2}{t_{1/2}} \quad (10)$$

Hence, the theoretical relative change in the mass concentration can be calculated and compared to the experimental calculated plots (**Figure 10**).

Based on the comparison in **Figure 10** it can be recognized, that the plots between the experimental and theoretical calculated relative change in mass concentration agree only for oleic acid. Therefore, the particle extraction process in partial recirculation operation of the transfer device with oleic acid as a surfactant can be approximately described as a reaction of 1st order. This indicates that the dissolution of oleic acid from the organic in the aqueous phase does not influence the process kinetics. On the contrary, the assumption of a 1st order reaction cannot be used to describe the process response if ricinoleic acid is applied as a surfactant. The course of the experimental relative change in the mass concentration resembles a s-shaped curve. Therefore to characterize the transfer kinetics we can use a sigmoid- or a logistic function, respectively, which is applied for approximating saturation processes and also dose-response-systems in Pharmacology [27]. Thus, the particular process behavior for ricinoleic acid can be described using the following equation:

$$\frac{\Delta\beta_A}{\beta_A} = f(t) = A_1 + \frac{A_2 - A_1}{1 + 10^{(M-t)*p}} \quad (11)$$

whereby A_1 and A_2 the theoretical lower and upper thresholds of the curve. Due to the fact that possible values for $\Delta\beta_A/\beta_A$ lie in the range $\{0...1\}$, A_1 and A_2 can be defined with 0 and 1, respectively. Furthermore, Equation (11) has a negative first derivation and, more important, one point of inflection, which demonstrates the local maximum of the mass flow at a process time between 4 - 5 minutes (**Figure 8**). The point of inflection is represented in Equation (11) by the parameter M , which is estimated through linear interpolation. The parameter p describes the slope of the curve and is numerically calculated by iteration. Thus, the function obtained is illustrated by the grey line in **Figure 10**, which accurately fits to the experimental data. This demonstrates that the additional mass transport due to dissolving of ricinoleic acid from the organic into the aqueous phase has an important influence on the kinetics of the particle extraction process within the column. The surfactant mass transfer can be assumed as the dose for the process, which predominates in the first minutes of the phase transfer when equilibrium of ricinoleic acid concentration in either phase is not reached. Consequently, the response is represented by the functionalization and subsequently the phase transfer of magnetite nanoparticles. Thus, it can be finally stated out that the particle extraction process is controlled by process imbalance.

4. Conclusions

In this work, the particle extraction process with a drop column as transfer device in partial recirculation operation has been investigated. The material parameter of the suspension are chosen to be constant with surfactant concentration $x_{\text{surf}} = 1.4$ mass-% and the optimal specific amount of surfactant per magnetite $X_{S/M} = 0.2$ g/g. For these parameters, secondary effects like water inclusion and emulsion formation can be excluded, because a stable process behavior with a complete clearing off the column as well as a sufficient coalescence rate can be achieved. Based on the modeling of the system, we are able to evaluate the transfer fluxes. Thereby, yields of transferred magnetite nanoparticles are obtained >80%. Furthermore, for the characterization of the process environment, we have chosen a particle-free phase transfer as an indicator. In combination with optical changes in the column as well as measurements of the total organic carbon (TOC) content and the size distribution using laser diffraction spectroscopy, we have proven that ricinoleic acid helps disintegrating iso-octane. Droplets with a median particle size of 5 - 10 μm are formed. This is particular difference in comparison to oleic acid and this is due to the chemical structure of the surfactants. Therefore, it is possible that additional phase interfaces are formed for the phase transfer process, which further determines the transfer times of particles by reduction of the interface tension. As a consequence of this, we have described the interaction of the processes and procedures at the phase boundary. Thereby, we have a mass transport of the surfactant from disperse into continuous phase on the one hand, which determines the process time for the phase transfer, and simultaneously the phase transfer of the magnetite nanoparticles on the other hand. This interconnection is reflected in the transfer kinetics of either surfactant. Finally, we have determined that the particle extraction process with oleic acid as a surfactant can be estimated as a reaction of 1st order. Thus, the influence of the surfactant mass transfer of the process kinetics is negligible. However, in the case of ricinoleic acid as surfactant, another approximation has to be used to describe the particular process behavior. This is represented by a sigmoid function in terms of a dose response curve. Therefore, the mass transport of the surfactant due to dissolving strongly influences the process kinetics.

Advanced studies in the process development of a continuous liquid-liquid phase transfer to obtain high-quality organosols will be presented soon regarding to produce stable colloids and interaction between the surfactants and organic phase used.

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