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Influence of Reaction Conditions on the Settling Behavior of Liquid-Liquid Dispersions

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DOI: 10.1002/cite.202000071

The settling behavior of liquid-liquid dispersions at ambient temperature and pressure is well investigated. However, little is known about the settling behavior of those systems at high pressure and high temperature. In this work, a novel stainless steel settling cell is presented, enabling investigations on liquid-liquid settling behavior at high pressures up to 130 bar. The settling behavior of a promising CO_2 hydrogenation reaction system is investigated by sequentially determining influences of dissolved CO_2, side components, and temperature.

Keywords: Batch settling experiments, Carbon capture and utilization, Gas-expanded liquids, Liquid-liquid dispersions, Temperature influence

Received: April 03, 2020; *revised:* May 22, 2020; *accepted:* June 18, 2020

1 Introduction

Using carbon dioxide (CO_2) as a feedstock for chemical processes, the so-called carbon capture and utilization (CCU), is a promising technology aiming to decrease the anthropogenic greenhouse effect. CO_2 from waste gas streams or directly from air is used as a C1 building block. By replacing intermediates refined from fossil resources, the resource efficiency of the carbon cycle in chemical synthesis can be improved. A possible reaction route is the already well investigated reaction of hydrogen (H_2) with CO_2 to formic acid (FA) [1–5]:

$$H_2 + CO_2 \rightleftharpoons \mathrm{FA} \tag{1}$$

This hydrogenation reaction can be homogeneously catalyzed by a liquid catalytic phase containing a dissolved ruthenium-based catalyst with bis(diphenylphosphino) methane ligands tailored with long alkyl chains [5]. A suitable solvent for the liquid catalytic phase is *n*-decane (Dec). The system is pressurized up to p_R = 135 bar enabling the solvation of the gaseous substrates H_2 and CO_2. Blast furnace gas in steel mills can be used as a feedstock for the substrates CO_2 and H_2. The addition of a second liquid phase, called product phase, enables an in situ extraction of the product FA by an amine-based product stabilizer dissolved in a suitable solvent. Thus, the conversion of the naturally endergonic reaction (ΔG^0 = 32.9 kJ mol^{-1}) is increased [1, 4]. Within the framework of the project Carbon2Chem, methanol (MeOH) was chosen as the solvent of the product phase and 1,2-dimethylimidazole (1,2-DMI) as the product stabilizer. In laboratory scale experiments with this specified system, high total turnover numbers up to 38 000 and turnover frequencies up to 1400 h^{-1} were achieved at mild reaction temperatures ($T_\mathrm{R} \approx$ 333–338 K) [5].

For continuous production of FA, the hydrogenation reaction can be performed in a continuously stirred tank reactor with dispersed liquid and gaseous phases. The outgoing stream is considered as a liquid-liquid dispersion and after the phase separation process, the catalytic phase can be recycled, while the product phase leaves the system for further purification or consecutive reactions [6].

Horizontal gravity settling is a possible unit operation for liquid-liquid phase separation. The required separators can be dimensioned with suitable models and knowledge of the phase separation behavior of the liquid-liquid dispersion at settling conditions [7]. The phase separation behavior of the liquid-liquid system can be characterized by batch settling experiments. The reaction mixture is therefore dispersed in a stirred tank in laboratory scale and the phase separation process is observed over time. The phase separation process depends on the sedimentation and coalescence behavior of the system. These phenomena are influenced by the volume fraction of the dispersed phase, the drop size distribution, and the physical properties of substances, e.g., density, viscosity, and interfacial tension. The drop size distribution results from the mixing process and, therefore, is dependent on the power input during the mixing process. The sedimentation process takes place immediately after the mixing process (Fig. 1). The sedimentation curve describes the boundary between liquid-liquid dispersion and coherent phase II.

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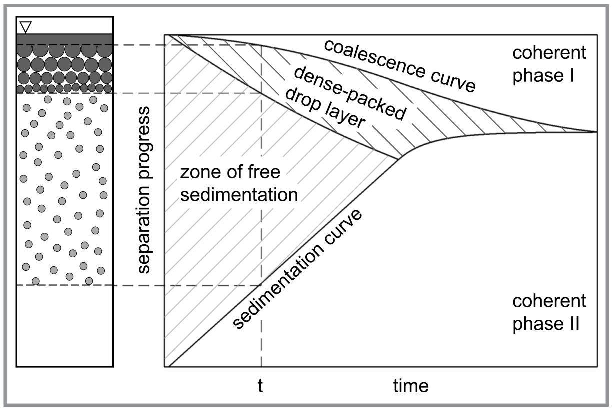

Figure 1. Separation process over time for liquid-liquid batch settling experiments [8] (modified). The light phase (coherent phase I) is dispersed in the heavy phase (coherent phase II).

When droplets accumulate at the liquid-liquid interface, a dense-packed drop layer is formed. The coalescence curve is determined by the merging of these drops and the interface to their coherent phase. Settling time, i.e., the time until the dispersion is completely separated into its coherent phases and a distinct phase boundary between both liquid phases remains, correlates with residence time of the reaction mixture in the gravity separator and thus its dimensions. Assuming constant horizontal flow velocity, the volume of the gravity separator has to be expanded with increasing settling time. To keep capital expenditures low, small gravity separators and, thus, short settling times are favorable.

Liquid-liquid settling processes are already well investigated for atmospheric pressure and ambient temperature [8–10]. Reaction temperature and pressure in chemical processes, however, mostly differ from atmospheric conditions. These influence the physical properties of the liquid-liquid system and, therefore, the phase separation behavior. At moderate pressure, Dec [11, 12] as well as MeOH [13, 14] dissolve large amounts of CO_2 which results in significant volume expansion of the liquid phases. The expanded liquid phases are so-called gas-expanded liquids [15] and due to phase expansion, a change in physical properties such as cross solubility, density, and viscosity of both solvents (MeOH and Dec) is caused. These physical properties are furthermore influenced by temperature and composition changes, e.g., by the presence of the product stabilizer 1,2-DMI.

Low capital and operational expenditures are basic requirements for designing a competitive CCU process. Therefore, not only small gravity separators are required, but the energy demand must also be kept as low as possible. If the liquid-liquid phase separation is carried out at reaction conditions, the energy demand for pressurization and heating of the recycled catalytic phase can be reduced.

To quantify the separation process of liquid-liquid dispersions at reaction conditions, a novel stainless steel settling cell was developed. In this work, the novel stainless steel settling cell that allows the investigation of the separation process of two liquid phases with the addition of gases is presented. The influence of reaction conditions on the settling behavior of a homogeneously catalyzed hydrogenation reaction system was studied neglecting influences of the homogeneous catalyst and dissolved H_2. Temperature influence was investigated by conducting settling experiments with the binary mixture MeOH and Dec at $T_1 \approx 293\,K$, $T_2 \approx 313\,K$, and $T_3 \approx 333\,K$. The influence of dissolved CO_2, FA, and 1,2-DMI on the separation process was investigated by considering different combinations of components and temperature influence.

2 Material and Methods

The presented settling cell (Fig. 2) with a total volume of $V_c = 1400\,cm^3$ was constructed according to a standardized batch settling cell diameter ($d_c = 80\,mm$) [7]. It was manufactured by Parr Instrument GmbH and is made from stainless steel T-316. To add gases to the liquid-liquid mixture, the cell is built with an increased height ($h_c = 220\,mm$) compared to the standardized cell. For optical access, it has two double sapphire sight glasses installed on opposite sides

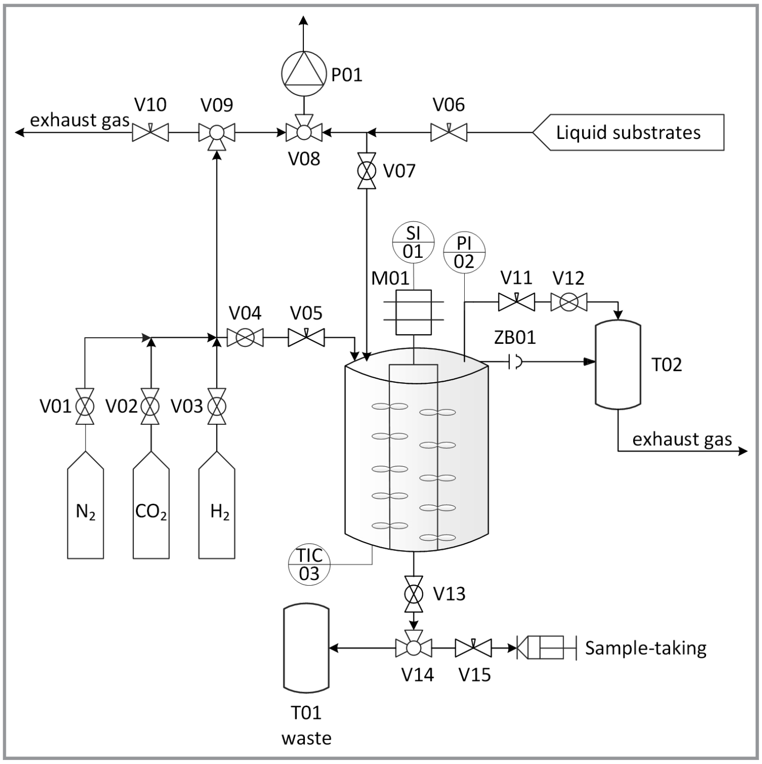

Figure 2. Experimental setup of the high-pressure settling cell.

of the cylinder surface, arranged at different heights and overlapping in the center. Each sight glass has a total height of h_w = 88.9 mm enabling visual observation of the settling behavior of the liquid-liquid dispersion over a total height of 165.1 mm. The inside of the stainless steel cell is illuminated by a cold light source with fiber-optic cable that is positioned at the bottom of the lower sight glass. Two Sony DCR-SR58E cameras are installed horizontally to record the settling progress (Fig. 3).

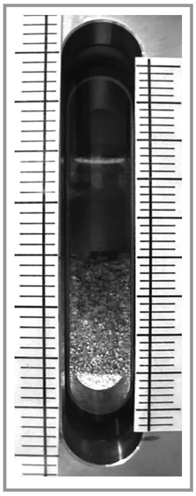

Figure 3. Double sapphire sight glass with the investigated system MeOH/Dec.

Inside the cell, two counter-rotating agitator shafts, each with five paddle stirrers attached, are located over the height of the liquid fluids. Counter bearings, positioned at the bottom, protect the agitator shafts against deflection. A Heidolph Hei-TORQUE Precision 100 stirrer motor (M01) drives the magnetic stirrer located at the top of the cell. The magnetic stirrer is connected to the agitator shafts through an internal gear. The cell is heated by two heating elements integrated in a double jacket system and cooled by cooling water supply. A PT-100 element positioned inside the cell measures the fluid temperature, which is controlled by a LTR2500 by Heju Juchheim Solingen controller (TIC03). The liquid components are added to the reactor via syringe. To prevent air from entering the cell after the filling process, the syringe connection is separated from the reactor by a manual ball valve (V07). The top of the cell has a connection to gas supply where nitrogen, H_2, and CO_2 can be added to the multiphase mixture with pressures up to p = 130 bar monitored by an S-11 WIKA pressure transmitter (PI02).

The investigated systems consist of MeOH with a purity of ≥ 99.9 % provided by VWR Chemicals BDH® and Dec with a purity of ≥ 99 % from Carl Roth GmbH + Co. KG. The product stabilizer 1,2-DMI was obtained from abcr GmbH with a purity of ≥ 98 % and FA was obtained from Merck KGaA with a purity of ≥ 98 %. CO_2 4.5 from AIR PRODUCTS was used. All chemicals were used as purchased without further purification.

A standardized experimental procedure was performed for all experiments. Therefore, the cell was first depressurized, and air was removed from the system by a laboratory vacuum pump (P01) until an absolute pressure of p = 0.1 bar was reached. The liquid phases were added to the system by syringe at room temperature and the syringe content was measured by an OHAUS NV2101 scale. Experiments were performed at a saturated liquid phase ratio of catalytic phase to product phase of ϕ = 0.44 (4.40 % standard deviation). To determine the initial composition of all liquid components and achieve the desired saturated phase ratio, preliminary experiments were conducted. Volume expansion was taken into account by adjusting the initial liquid filling volume and all experiments were performed with a filling height of 179.5 mm (6.16 % standard deviation) in vapor-liquid-liquid equilibrium. The results are presented in relation to the relative height; the surface of the liquid-liquid system corresponds to a relative height of 1.

To reach thermodynamic equilibrium at a given temperature, temperature control was started, and the stirring motor was set to 400 rpm. If experiments were performed with CO_2, the system pressure was kept constant by continuously adding the gaseous component during the saturation process. When vapor-liquid-liquid equilibrium was reached, stirring velocity was increased to 800 rpm for t = 60 s. Stirring motor and temperature control were then switched off to record the settling behavior of the system by video camera. The temperature control had to be switched off because strong temperature gradients influenced the local solubility, which was shown by streaking. The reasons for the temperature gradients are lack of mixing combined with the position of the heating elements and the PT-100 element. When temperature control was switched off during the settling experiment, temperature did not significantly change. Each experiment was carried out three times and the recordings of the cameras were optically analyzed afterwards.

The reference test system of the settling experiments at $T_1 \approx 293$ K consists of MeOH, the main component of the heavy phase, and Dec, the main component of the light phase. Since cross solubility of MeOH and Dec increases with increasing temperature, the influence of temperature on the liquid-liquid system was investigated by performing settling experiments with this binary system at $T_2 \approx 313$ K and $T_3 \approx 333$ K.

FA and the product stabilizer 1,2-DMI affect the properties and composition of the liquid phases. The settling behavior of the quaternary system MeOH, Dec, 1,2-DMI, and FA was investigated at the same three temperatures as the binary reference system. Experiments without the addition of CO_2 were performed at the vapor pressure of the mixture. To investigate the impact of a dissolved gas on the settling behavior, CO_2 was added to both the binary system and the quaternary system until a total pressure of $p \approx 30$ bar was reached. Both CO_2-pressurized systems were examined at the three temperatures T_1–T_3 since the solubility of CO_2 in the main components MeOH and Dec strongly correlates with temperature.

3 Results and Discussion

All liquid-liquid settling experiments were performed with the respective phase ratio of $\phi \approx 0.44$ and reveal that the Dec-rich phase is dispersed in the continuous MeOH-rich phase. An overview of settling times for all measured systems at different temperatures is given in Tab. 1. The different settling trends with rising temperature are discussed below. A settling time of only t = 17.9 s at T_1 = 295.03 ± 0.01 K (Tab. 1) with no coalescence inhibition and fast sedimentation velocities is detected for the binary reference system MeOH and Dec (Fig. 4).

Table 1. Settling times (in s) of the measured systems at different temperatures.

Components	293 K	313 K	333 K
MeOH-Dec$^{a)}$	17.9	16.3	21.7
MeOH-Dec-FA-DMI$^{a)}$	59.9	39.6	33.9
MeOH-Dec-$CO_2$$^{b)}$	20.5	23.5	42.4
MeOH-Dec-FA-DMI-$CO_2$$^{b)}$	49.1	41.1	28.5

a) Measurement at vapor pressure; b) measurement at $p \approx 30$ bar.

A temperature increase of ΔT = 20 K has a positive impact on phase separation time, which is in accordance with literature [16, 17]. With further increase in temperature to T_3 = 334.50 ± 0.34 K, settling time increases by 33.1 % compared to T_2. Hence, results contradict previous observations of temperature-dependent settling behavior at constant stirrer speed. The sedimentation velocity of droplets is proportional to the initial slope of the sedimentation curve [7]. Thus, the results reveal that sedimentation velocity remains the same for the two temperatures T_1 and T_2, while the sedimentation velocity decreases for T_3. While bigger droplets sediment faster, a higher viscosity of the continuous phase and a smaller density difference result in slower sedimentation.

Drop size distribution depends on physical properties, e.g., viscosity and interfacial tension, and operating parameters such as the energy input into the system through the prior mixing process [18]. For both pure liquid components, the absolute difference in density and the viscosity of MeOH, the main component of the continuous phase, decrease over temperature (Fig. 5). Since stirring velocity stays constant over the whole temperature range, different drop size distributions must result. Miscibility effects of dissolved components must be considered though to predict sedimentation behavior by physicochemical substance properties. With the increase in temperature, density difference between the continuous and dispersed phase decreases due to higher miscibility of MeOH in Dec. While only 5.6 mol % MeOH dissolve in Dec at T = 290.24 K, the solubility of MeOH in Dec increases to 25.44 mol % at T = 334.95 K [21]. In contrast, the solubility of Dec in MeOH is not heavily temperature-dependent in the temperature interval considered. The solubility increases only from 2.19 mol % Dec dissolved in MeOH at T = 290.98 K to 5.13 mol % at T = 333.67 K [21]. This leads to the slower sedimentation velocity for T_3. As the sedimentation velocity is equal for T_1 and T_2, the decrease in settling time for T_2 is caused by a different coalescence behavior.

The phenomenon of cross solubility becomes more visible with the addition of CO_2, which promotes the solubility of MeOH in Dec and vice versa. In contrast to the binary test system, the two temperature steps investigated with the addition of CO_2 both result in longer settling times (Fig. 6). Furthermore, the absolute settling time increases for each examined temperature point (Tab. 1) and, thus, the addition of CO_2 negatively influences the settling behavior by increasing the separation time. A high increase in separation time by factor 2.0 in relation to the test system without CO_2 (Fig. 4) is observed for the ternary test system of MeOH, Dec, and CO_2 at p_3 = 29.3 ± 0.05 bar and T_3 = 334.64 ± 0.37 K (Fig. 6). With the addition of CO_2, the physicochemical properties of the two liquid phases converge because the upper critical solution temperature (UCST) of the system is lowered resulting in slower sedimentation velocities of the Dec-rich phase for each investigated temperature. However, since the solubility of CO_2 in pure MeOH and in Dec decreases with increasing temperature, both phases contain different amounts of

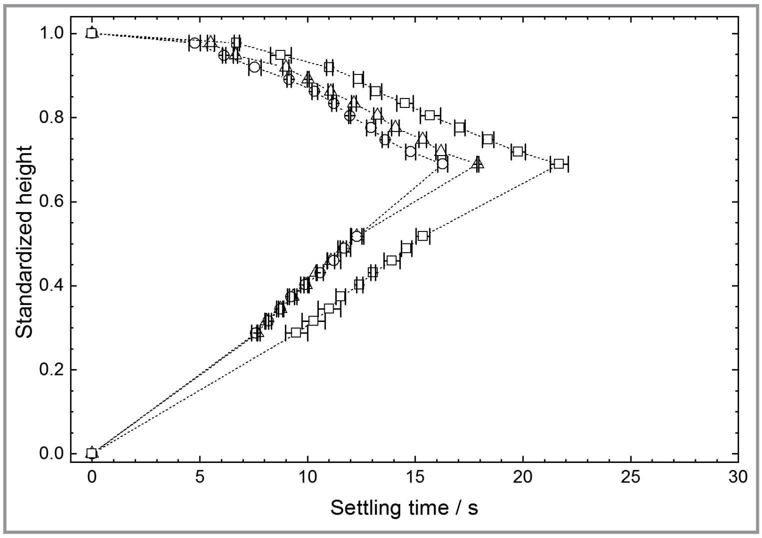

Figure 4. Settling behavior of the binary test system MeOH and Dec at T_1 = 295.03 ± 0.01 K (△), T_2 = 314.82 ± 0.24 K (○), T_3 = 334.50 ± 0.34 K (□).

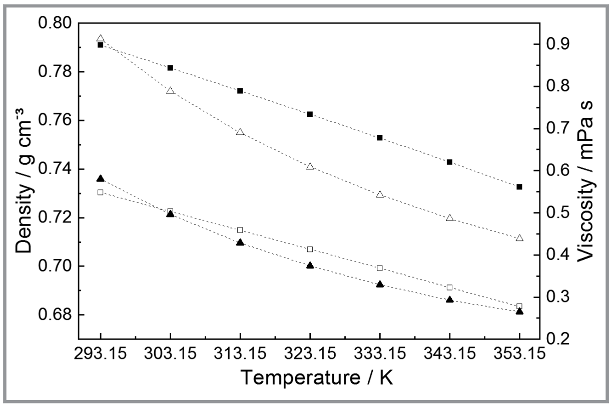

Figure 5. Physicochemical property change of pure liquids over temperature: density of MeOH [19] (■) and Dec [19] (□), viscosity of MeOH [20] (▲) and Dec [19] (△).

dissolved CO_2 for each temperature [10–13]. Thus, physical properties of the ternary component system are not only affected by temperature influences, but also by the absolute component composition of the two liquid phases. However, it must be noted that the absolute settling times of the investigated liquid-liquid system are still short and there is no inhibition of coalescence.

For all quaternary and quinary experiments performed, starting composition for the product phase is 12.30 mol % FA, 35.00 mol % 1,2-DMI, and 52.70 mol % MeOH. The addition of FA and 1,2-DMI to the binary reference system increases settling time at $T_1 \approx 293$ K with a factor of 3.4 (Tab. 1). Additionally, temperature-dependent experimental results for the quaternary system show a decrease in settling time with increasing temperature (Fig. 7). In particular, the sedimentation velocity increases with rising temperature within the investigated temperature range. The addition of both FA and 1,2-DMI leads to changes in the physicochemical properties of the mixture, but also to a higher polarity of the product phase and, thus, to an increase in the UCST. When dispersing the liquids at constant stirrer velocity, the torque is proportional to the viscosity of the liquid phase. When viscosity of the liquid phase decreases because of increasing temperature, bigger droplets result due to the decreasing torque value. For this system, the positive effect of the temperature increase, and thus, bigger droplets on the settling behavior can be observed for the whole temperature range.

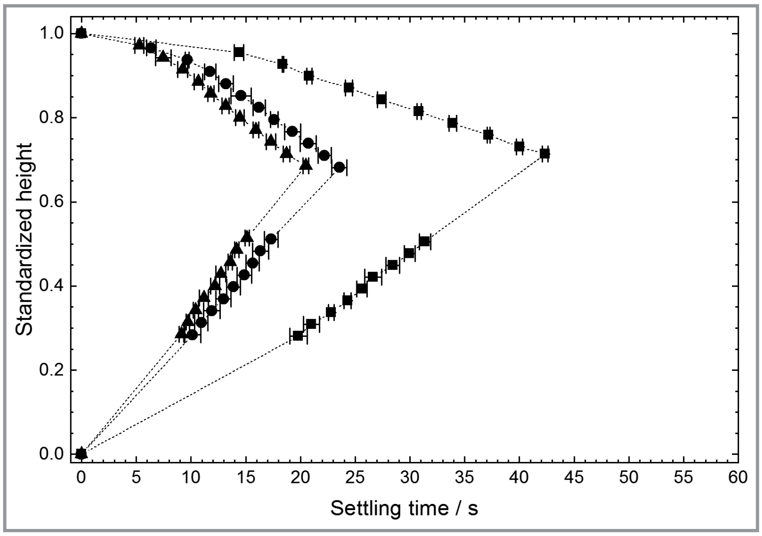

Figure 6. Settling behavior of the pressurized test system MeOH, Dec, and CO_2 at $T_1 = 295.05 \pm 0.01$ K and $p_1 = 30.1 \pm 0.05$ bar (▲), $T_2 = 314.79 \pm 0.25$ K and at $p_2 = 30.0 \pm 0.08$ bar (●), $T_3 = 334.64 \pm 0.37$ K and $p_3 = 29.3 \pm 0.05$ bar (■).

Furthermore, the addition of CO_2 to the quaternary liquid-liquid system with a system pressure of $p = 29.74 \pm 0.23$ bar has only minor influences (Tab. 1). The settling time is in the same range for each temperature point as for the quaternary reference system and decreases with increasing temperature (Fig. 8). This means that the negative influence of CO_2 on the sedimentation behavior of the binary test system cannot be reproduced for the quaternary system. On the other hand, when adding CO_2 to the quaternary system, the volume expansion of both liquid phases indicates that significant amounts of CO_2 are still soluble. When comparing sedimentation velocities, the Dec-rich phase sediments faster after the addition of CO_2. Again, droplet size, density, and viscosity changes can lead to the changes in sedimentation velocity. Due to missing physicochemical property data, no statement on density and viscosity with temperature dependency can be made. To theoretically describe density and viscosity of this system with and without the addition of CO_2, the exact molar fraction of all components in each phase needs to be determined. Consequently, either a theoretical model, describing temperature dependency, viscosity, and interfacial tension for this temperature and pressure range needs to be developed or physicochemical experimental data needs to be generated.

Overall, the experimental results confirm that the construction of the presented stainless steel cell enables the investigation of sedimentation and coalescence with limitations. No settling behavior up to 30 % of relative height can be observed because of the limited length of the sight glass. This limited length of the sight glasses is caused by the required pressure resistance for pressures up to $p = 130$ bar. In the height, where the sight glasses are exactly opposite, the scattering of light and large camera angles lead to uncertainty. Thus, settling progress is depicted in the range of 25.8–51.7 % and of 61.9–100 % of the relative height in Fig. 4 and Figs. 6–8, respectively.

When transferring the settling behavior results to process and reactor design, the decrease in separation time for the quinary system at high temperature leads to lower investment costs for

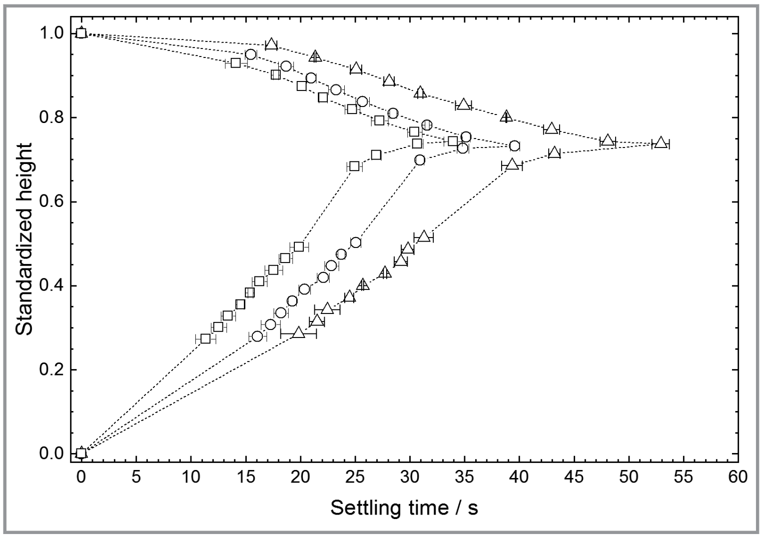

Figure 7. Settling behavior of the quaternary test system MeOH, 1,2-DMI, FA, and Dec at $T_1 = 295.79 \pm 0.02$ K (△), $T_2 = 312.68 \pm 0.44$ K (○), $T_3 = 333.02 \pm 0.29$ K (□).

gravity separators. Furthermore, by separating the system at reaction temperature, heating of the recycled Dec-rich phase before entering the hydrogenation reactor is avoided. This results in a reduction of the overall process energy demand. Regarding the influence of CO_2 on the separation behavior of the quinary system, the results of this study show that the reaction mixture can be separated without prior pressure relief. While a separation of CO_2, MeOH, and Dec at T_3 and $p \approx 30$ bar results in a 2.4 times higher settling time compared to the binary reference system at T_1, the settling time of the quinary system was decreased by a factor of 2.1 at $p \approx 30$ bar and T_3 in relation to the quaternary reference system at T_1.

Thus, for the homogeneously catalyzed hydrogenation, a phase separation at reaction conditions should be carried out, when neglecting the presence of H_2. With this system, neither dissolved CO_2 nor temperature influences hinder coalescence or decrease sedimentation velocity. However, the results of the presented case studies show the need to investigate the influence of the reaction conditions on the settling behavior for each new system. No general conclusion can be drawn as to whether systems should be separated at reaction conditions. The liquid-liquid settling behavior and, thus, the design of gravity phase separators depend on physicochemical phase data such as viscosity, density, and interfacial tension, which are affected to varying degrees by highly soluble gases and temperature.

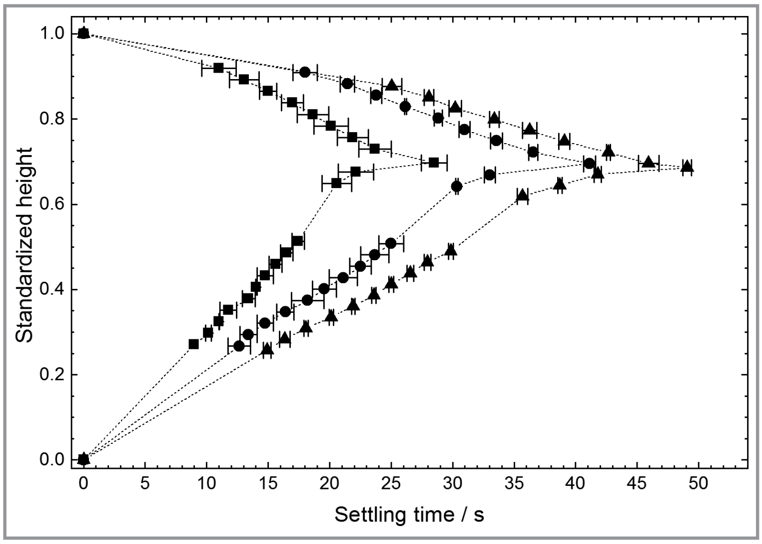

Figure 8. Settling behavior of the pressurized test system MeOH, 1,2-DMI, FA, Dec, and CO_2 at $T_1 = 295.79 \pm 0.01$ K and $p_1 = 29.5$ bar (▲), $T_2 = 313.75 \pm 0.30$ K and $p_2 = 30.0 \pm 0.1$ bar (•), $T_3 = 332.56 \pm 0.40$ K and $p_3 = 29.7 \pm 0.11$ bar (■).

4 Conclusion

A novel high-pressure settling cell which enables the investigation of the settling behavior of liquid-liquid dispersions up to pressures of $p = 130$ bar was presented. Influences of temperature and CO_2 solubility were examined in two case studies: the binary component system MeOH/Dec and the quaternary component system MeOH/Dec/1,2-DMI/FA. Both test systems were investigated at saturated vapor pressure and at a total pressure of $p \approx 30$ bar. For the quaternary and quinary test system, temperature increase resulted in decreasing settling time. This effect was reproducible for a temperature increase from $T_1 \approx 293$ K to $T_2 \approx 313$ K for the binary test system MeOH and Dec. After all, for this system, a further rise in temperature up to $T_3 \approx 333$ K resulted in an increase of the settling time. Settling time of the ternary system MeOH, Dec, and CO_2 increased for all investigated temperature steps.

While the addition of CO_2 has a negative effect on the settling behavior of the binary test system MeOH and Dec, no such trend was found in presence of FA and 1,2-DMI. The addition of CO_2 increases the cross solubility of MeOH and Dec in relation to the binary component mixture, thus, changing densities, viscosities, and interfacial tension of the liquid-liquid system. This implies a decrease of the UCST and an increase of the settling time in the investigated temperature range. The further components 1,2-DMI and FA decrease the cross solubility of MeOH and Dec and mainly stay in the MeOH-rich product phase [5]. The addition of

CO_2 had only a minor influence on settling time indicating that CO_2 in the quinary mixture does not affect the physicochemical properties as much as in the ternary mixture.

The effects of the reaction conditions, i.e., temperature influence and influence of dissolved gases, on the settling behavior of liquid-liquid dispersions outlined in this article reveal that there is no general conclusion deducible from results whether gravity separation should be carried out at these conditions. Physicochemical phase data that influences the liquid-liquid gravity settling time is affected by both temperature and addition of CO_2. For the liquid component system MeOH/Dec/FA/1,2-DMI, the settling time was reduced for experiments at $T_3 \approx 333\,K$ in presence of CO_2 compared to quaternary experiments at $T_1 \approx 293\,K$. By omitting the pressurization and heating of the recycled catalytic Dec-rich phase before it is returned to the hydrogenation, the energy demand of the overall process is favored. For the holistic design of gravity separators, physicochemical phase properties must be known [7], which requires further experimental studies or theoretical models to describe the physicochemical phase properties of the investigated system at reaction conditions.

The authors gratefully acknowledge funding for the Carbon2Chem project (03EK3042C) by the German Federal Ministry of Education and Research (BMBF). Additional financial support by the NAMOSYN project (03SF0566P0) funded by the BMBF is gratefully acknowledged. Open access funding enabled and organized by Projekt DEAL.

Symbols used

d	[mm]	inner diameter
ΔG^0	[kJ mol^{-1}]	Gibbs free energy
h	[mm]	height
p	[bar]	pressure
t	[s]	time
T	[K]	temperature
V	[cm^3]	volume

Greek letter

ϕ	[–]	phase ratio

Subscripts

c	cell
R	reaction
w	double sapphire sight glass

Abbreviations

CCU	carbon capture and utilization
Dec	*n*-decane
1,2-DMI	1,2-dimethylimidazole
FA	formic acid
MeOH	methanol
UCST	upper critical solution temperature

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DOI: 10.1002/cite.202000071

Influence of Reaction Conditions on the Settling Behavior of Liquid-Liquid Dispersions

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Research Article: Concerning process design, it can be advantageous to separate the phases of a multiphase reaction system at reaction conditions. In this work, the settling behavior of liquid-liquid dispersions is investigated at reaction conditions. For this purpose, a new batch settling cell was developed that is both high temperature- and pressure-resistant. .... ■