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# Time-dependent structural breakdown of microencapsulated phase change materials suspensions

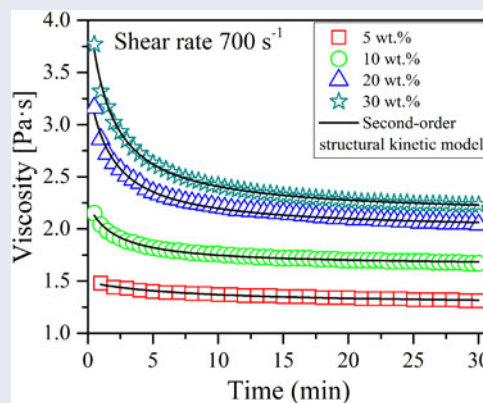
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## ABSTRACT

Microencapsulated phase change materials (MPCM) suspensions are multi-phase heat transfer fluids which exploit the latent heat of phase change materials. The effect of MPCM on the rheological properties of suspensions of microcapsules in glycerol were investigated to explore the suitability of the suspensions as a pumpable heat transfer fluid. Three different rheological models were utilized to characterize the time-dependent structural breakdown of the suspensions, and the second-order structural kinetic model was found to give a better fit to the experimental data than the Weltman and Fignon-Shoemaker models. The MPCM form agglomerates, which are disrupted by shear forces. The breakdown of the agglomerated structures was most pronounced at high shear rates where the microcapsules are subjected to stronger disruptive forces. More agglomerates are present at higher concentrations, which causes a stronger breakdown of the agglomerated structures when the concentration is raised. The time-dependent structural breakdown of MPCM suspensions plays an important role for improving the efficiency of heat transfer liquids based on such materials.

## GRAPHICAL ABSTRACT



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## KEYWORDS

Microencapsulated phase change materials; rheology; the second-order structural kinetic model; time-dependent behaviour

## Introduction

Suspensions of microcapsules in heat transfer fluids have great potential for thermal energy storage and heat transfer fluid applications.<sup>[1–6]</sup> Microencapsulated phase change materials (MPCM) suspensions are multi-phase heat transfer fluids which exploit the latent heat of phase change materials. They are more efficient heat carriers than single-phase fluids. An increase in MPCM concentration improves the heat capacity and energy storage density during the phase

change temperature range. Accordingly, reduced suspension flow rates can be utilized, leading to a lower pumping power consumption. However, this is counteracted by the MPCM induced viscosity increase of the suspensions, which raises the power consumption for pumping. It is therefore important to examine the rheological properties of the MPCM suspensions. Most studies of MPCM suspensions are only considering Newtonian liquids.<sup>[2–5]</sup> However, many such systems exhibit non-Newtonian and time-dependent behavior.

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Water is the most utilized fluid for microcapsule suspensions due to its availability, cheap price, high thermal conductivity and large specific heat capacity.<sup>[2–7]</sup> The main problems of water based MPCM suspensions are the high floatation rate and the restricted usable temperature range. In addition, agglomeration of the microcapsules can cause problems such as increased viscosities. The agglomeration of the microcapsules can be reduced by utilizing a reasonable microcapsule concentration<sup>[8]</sup> and by using surfactants.<sup>[3,6]</sup> Flotation can be averted by using smaller microcapsules<sup>[8]</sup> and by balancing the density of the microcapsules and the carrier medium. In addition, a fluid with a higher viscosity, such as glycerol, can also reduce floatation. Glycerol exhibits a high thermal conductivity and large specific heat capacity, and a lower freezing point and higher boiling point than water. This can extend the usable temperature range. However, the main drawback of utilizing glycerol as a carrier fluid is the higher viscosity, which increases the power consumption of the pumping process. It is therefore interesting to investigate the effect of MPCM on the rheological properties of glycerol suspensions.

In this study, the time-dependent structural breakdown of microcapsule suspensions in glycerol under the influence of a steady shear rate was investigated. The time-dependent behaviour of the MPCM suspensions were fitted to three different models in order to find which model is best suited to describe the experimental data (the Weltman model,<sup>[9]</sup> the Fighi & Shoemaker model<sup>[10]</sup> and the second-order structural kinetic model).<sup>[11,12]</sup> The investigation provide valuable rheological information which can be used to minimize the effect of MPCM on the viscosity increase of the suspensions. This can help enhancing the energy efficiency of this new type MPCM suspension.

## Materials and methods

The microencapsulated phase change materials (MPCM) was made by a spray drying process.<sup>[13]</sup> The MPCM is composed of a paraffin Rubitherm®RT27 core coated with a LDPE-EVA (low density polyethylene (LDPE) and ethylvinylacetate (EVA) copolymer) shell<sup>[13]</sup> The diameters of the single microcapsules are about 3–10  $\mu\text{m}$  (SEM image-Figure 1a). However, the microcapsules are prone to agglomeration with an agglomerated particle size distribution (Malvern

MasterSizer-UK) in the range of 10–550  $\mu\text{m}$  with a median value of 170  $\mu\text{m}$  (50% in the cumulative distribution) (Figure 1b).

The LDPE-EVA/RT27 has a high latent heat of 110 J/g and a melting point of 27 °C. MPCM suspensions were fabricated by dispensing different mass ratios of MPCM in glycerol. The mass concentration was varied from 0 to 30 wt.%.

## Thermal properties of MPCM suspensions

The latent heat of MPCM suspension was determined using a Mettler Toledo DSC822e fitted with a MultiSTAR HSS7 sensor, under an inert atmosphere. The heating rate for this process was set at 5 °C/min. The latent heat of the MPCM suspensions were determined over the range of –20 °C to 40 °C.

## Time-dependent structural breakdown of MPCM suspensions at constant shear

Time-dependent structural breakdown of MPCM suspension were carried out using an Anton Paar MCR301 rheometer (Austria). The MPCM suspensions were tested using a CC27 bob/cop measuring system (cup diameter: 28.91 mm; bob diameter: 26.66 mm) mounted in a cylindrical Peltier system for temperature control. A fresh sample was loaded into the measuring system, pre-sheared at shear rate of 50  $\text{s}^{-1}$  for 5 min, and rested for 5 min before any measurements were conducted. A pre-test (data not shown) confirmed that a 5 min resting time was long enough to reach a steady state.

The time-dependent behaviour of the MPCM suspensions were investigated during a period of 30 min at 20 °C with a constant shear rate of 100, 300, 500 and 700  $\text{s}^{-1}$ . In order to investigate the reproducibility of the results, each measurement was repeated three times with fresh samples. Experimental data were fitted to three different models: the Weltman model, the Fighi-Shoemaker model and the second-order structural kinetic model.

The Weltman model (Equation 1) is used to determine the logarithmic decrease of the shear stress with shearing time. The Weltman model includes two main parameters to estimate the time-dependent behaviour of suspensions.  $\sigma_0$  represents the initial shear stress needed to start degrading the structure of the material. The quantity of structure

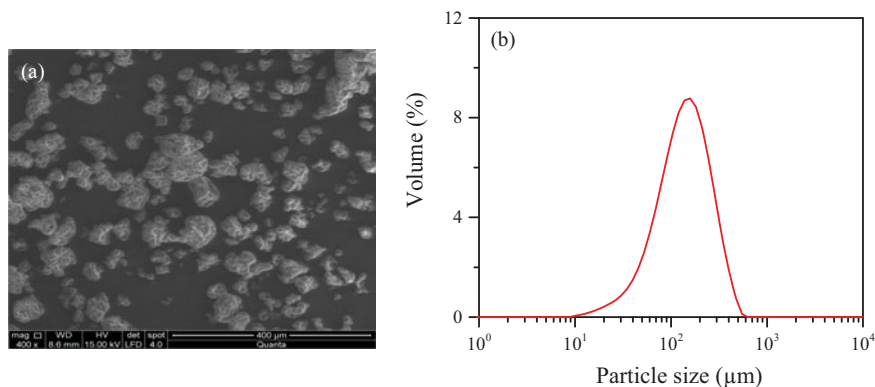


Figure 1. (a) SEM image and (b) size distribution of LDPE-EVA/RT27 microcapsules.

degradation during shearing is estimated by the time coefficient of breakdown (B). This also indicates the reduced rate of shear stress from the initial value to the final equilibrium value.<sup>[14]</sup>

$$\sigma(t) = \sigma_0 - B \ln(t) \quad [1]$$

The Figoni-Shoemaker model (Equation 2) describes the time-dependent behavior in a kinetic constant of breakdown of the internal structure of the MPCM suspensions. This model allows the quantification of the remaining structure and the structure breakdown by the parameters  $\sigma_e$  and  $(\sigma_0 - \sigma_e)$ , respectively.

$$\sigma_t = \sigma_e + (\sigma_0 - \sigma_e) \exp(-kt) \quad [2]$$

where  $k$  is the kinetic constant of structural breakdown.

Finally, the second-order structural kinetic model assumes that the change of structure is associated with breakdown of the internal fluid structure during the shearing process. The kinetics of the structured state to non-structured state process will define the structural breakdown rate during shearing process.<sup>[12,15]</sup>

$$\left( \frac{\eta - \eta_e}{\eta_0 - \eta_e} \right)^{1-m} = (m-1)k_s + 1 \quad [3]$$

where  $\eta_0$  is the initial viscosity at  $t=0$  (structured state),  $\eta_e$  is the equilibrium viscosity as  $t \rightarrow \infty$  (non-structured state),  $m$  is the order of the structure breakdown reaction,  $m=2$  for second-order structural kinetic model.<sup>[12]</sup>

The model selection for characterizing the time-dependent behaviour of the MPCM suspensions was evaluated using the determination coefficient  $R^2$  and the normalized root mean squared error (NRMSE).

## Results and Discussion

Figure 2 shows the DSC thermograms of the MPCM suspensions at different concentrations of MPCM. There are two distinct DSC peaks in Figure 3a: the main peak (10–40 °C) which represents the melting range of the paraffin Rubitherm® RT27 core, and a minor peak (0–5 °C) which corresponds to the melting of water. Figure 2b illustrates the latent heat of the MPCM suspensions. As expected, the latent heat of the suspensions is directly proportional to the

MPCM concentration. After adding 30 wt.% of MPCM, the latent heat is approximately 27 J/g.

The time-dependent behaviour of the MPCM suspensions were studied at constant shear rates of 100, 300, 500 and 700  $s^{-1}$  at 20 °C for a period of 30 min at different microcapsule concentrations. The experimental data was fitted to three commonly used models (the second-order structural kinetic model, the Weltman model and the Figoni-Shoemaker model) to investigate which of these models describes the time-dependent behaviour of the MPCM suspensions best.

Figure 3 illustrates that the experimental data fits reasonably well to the Weltman and Figoni-Shoemaker models, although there are some clear deviations at short times for some of the samples, especially for high concentrations and at high shear rates. As can be seen from Figure 4, the parameters  $\sigma_0$  and  $B$  from the Weltman model exhibit higher values with increasing shear rates and concentrations.  $\sigma_0$  is related to the shear stress at zero time, and becomes higher as the concentration is increased due to the higher viscosity of the samples, and will naturally increase at higher shear rates. When the microcapsule suspensions are subjected to a constant shear rate during the 30 min rheological experiment, the microcapsule agglomerates are gradually broken down by the shear forces. This structure break-down is quantified by the parameter  $B$ . As expected, more of the agglomerates are broken down when the samples are subjected to higher shear rates (higher values of  $B$ ). In addition,  $B$  increases when the microcapsule concentration is raised. This is due to the enhanced tendency to form aggregates at higher concentrations. Accordingly, there are more agglomerates to break down, which causes higher values of  $B$ . Similar trends were also reported by Durairaj et al.<sup>[16]</sup>

Figure 5 shows the parameters from the fits to the Figoni-Shoemaker model, where all parameters increase when the shear rates and concentrations are raised. The initial shear stress ( $\sigma_0$ ) (Figure 5a) have values close to that obtained from the Weltman model (Figure 4a). The equilibrium shear stress ( $\sigma_e$ ) (Figure 5b) increases with concentration due to the viscosity increase caused by more microcapsules in the suspension. The quantity of structure breakdown ( $\sigma_0 - \sigma_e$ ) (Figure 5c) becomes higher when the microcapsule concentration is raised, since there are more aggregates present that can be broken down by the

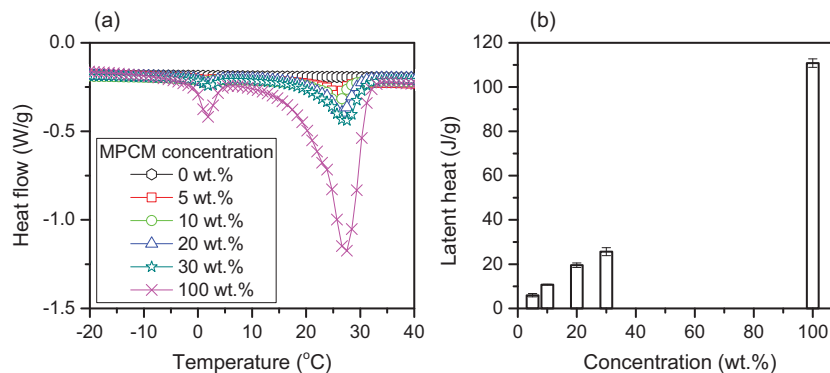
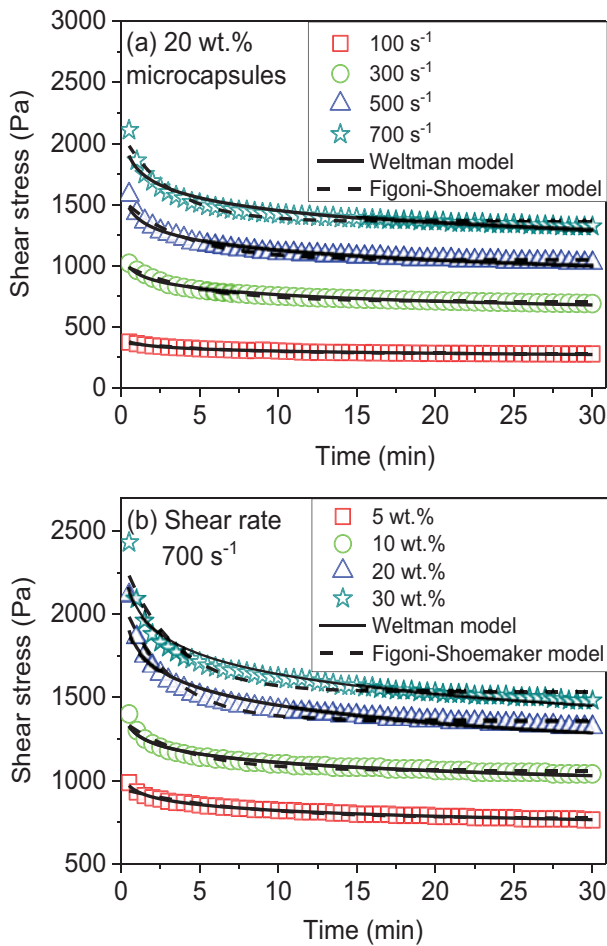
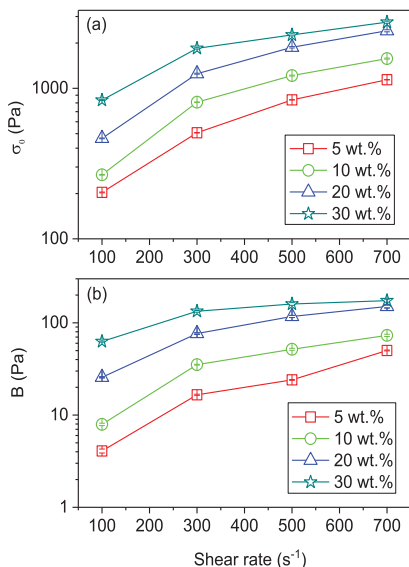


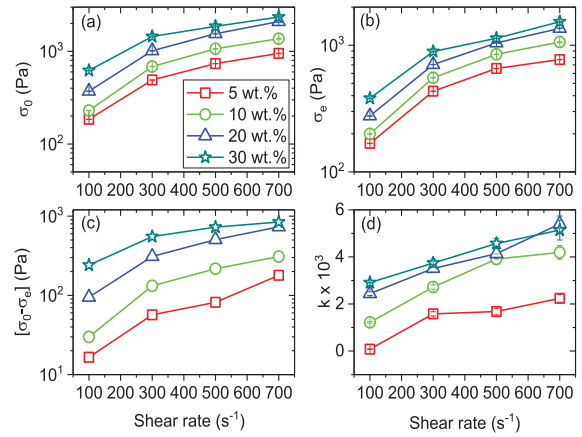
Figure 2. (a) The heat flow as function of temperature and (b) the latent heat of the MPCM suspensions at different concentrations of microcapsules.



**Figure 3.** Shear stress at (a) different shear rates, MPCM concentration 20 wt.%, and (b) different concentrations, shear rate  $700 \text{ s}^{-1}$  as a function of shearing time at  $20^\circ\text{C}$ . The points are experimental values. The solid and dashed lines show the fitted values according to the Weltman model (Equation 1) and the Fighi-Shoemaker model (Equation 2), respectively.



**Figure 4.** Rheological parameters of MPCM suspensions as a function of shear rates and concentration obtained by the Weltman model (Equation 1) at  $20^\circ\text{C}$ , (a)  $\sigma_0$  and (b)  $B$ .



**Figure 5.** Rheological parameters of MPCM suspensions as a function of shear rates and concentration obtained by the Fighi-Shoemaker Model (Equation 2) at  $20^\circ\text{C}$ , (a)  $\sigma_0$ , (b)  $\sigma_e$ , (c)  $[\sigma_0 - \sigma_e]$  and (d)  $k$ .

shear stress. In addition, higher shear rates breaks down more of the aggregates. The kinetic constant of structural breakdown ( $k$ ) illustrates how fast the stress of the MPCM suspensions reaches equilibrium under a constant shear. As can be seen from Figure 5d, the systems are approaching equilibrium conditions faster at higher shear rates, where the agglomerates are broken down more quickly. This effect has also been reported previously for other systems.<sup>[14]</sup> It is interesting that an increasing concentration of the microcapsules also causes a faster approach to the equilibrium values. It is possible that the closer proximity of the agglomerates to each other speed up the process due to more frequent collisions between the agglomerated structures.

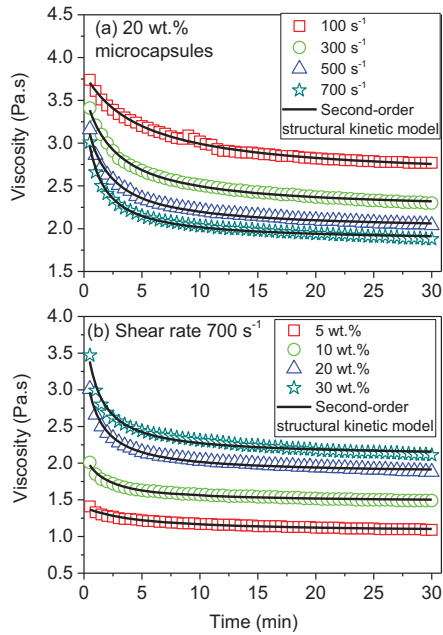
The rate constant ( $k_s$ ), of the second-order structural kinetic model (Equation 3) probes the kinetics of the breakdown of structures in the liquid, while the ratio of initial to equilibrium viscosity ( $\eta_0/\eta_e$ ) is related to the extent of structural decay due to the shear forces.<sup>[12,15,17]</sup> Figure 6 shows the fitted data curves of the MPCM suspensions using the second-order structural kinetic model. The model seems to fit well with the experimental data at all conditions. At a constant shear rate, the viscosity decreases drastically with time in the first 5 min, before approaching a plateau region after approximately 15 min. This illustrates that the MPCM agglomerates are disrupted by the shear forces. Hammadi et al.<sup>[18]</sup> and Mallik et al.<sup>[19]</sup> observed similar trends for clays suspension and solder paste suspensions, respectively.

The viscosity decays more rapidly toward an equilibrium viscosity when the shear rates is raised (Figure 6a), illustrating that high shear rates accelerate the breakdown of the MPCM agglomerates. In addition, the rate constant ( $k_s$ ) becomes higher with increasing shear rate and concentration (Figure 7c), revealing that the degradation rate of MPCM suspensions increases with increasing shear rates and concentrations. This is analogous to what was observed for he kinetic constant of structural breakdown ( $k$ ) in the Fighi-Shoemaker model (Figure 5d).

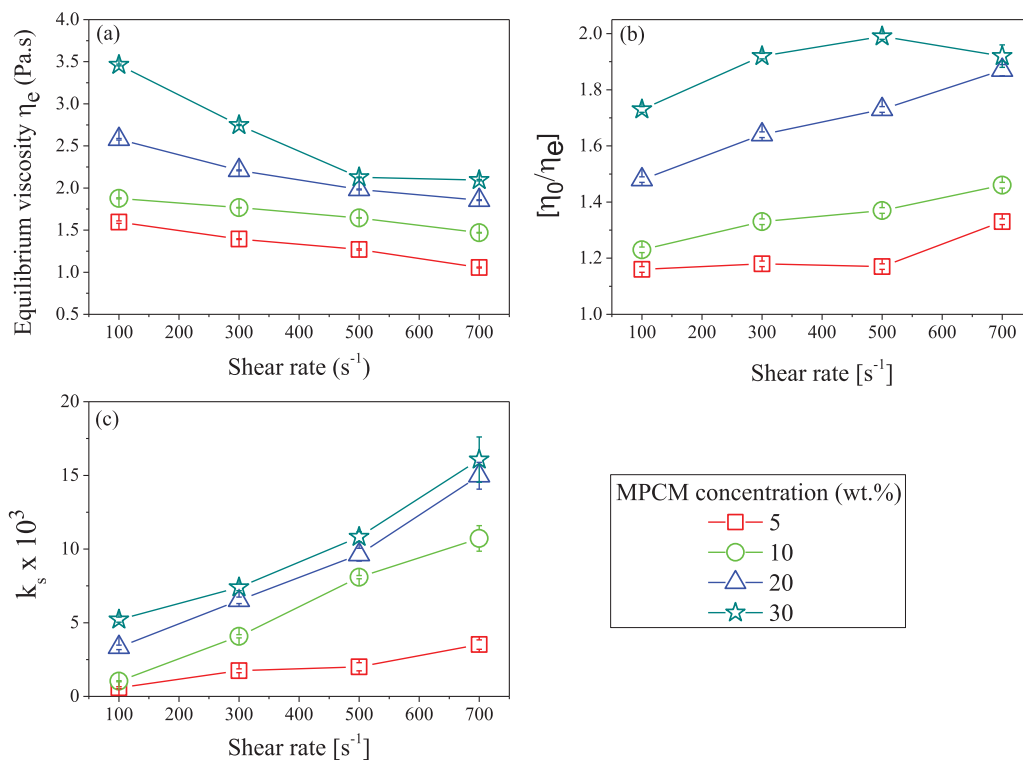
The equilibrium viscosity ( $\eta_e$ ) in Figure 7a illustrates the viscosity at long times where the agglomerates are broken down by the applied shear rate. As expected, the equilibrium viscosity becomes higher when the MPCM concentration

increases due to the presence of more particles. When the shear rate is raised,  $\eta_e$  decreases since higher shear forces can break the agglomerates down to smaller structures. This illustrates the importance of avoiding large agglomerates for applications where a low viscosity fluid is preferred.

Figure 7b shows that the amount of structural breakdown ( $\eta_0/\eta_e$ ) rises when the shear rate and the concentration are



**Figure 6.** Viscosity at (a) different shear rates, MPCM concentration 20 wt.%, and (b) different concentration, shear rate  $700 \text{ s}^{-1}$  as a function of shearing time at  $20^\circ\text{C}$ . The points are experimental values. The solid lines show the fitted values according to the second-order structural kinetic model (Equation 3).

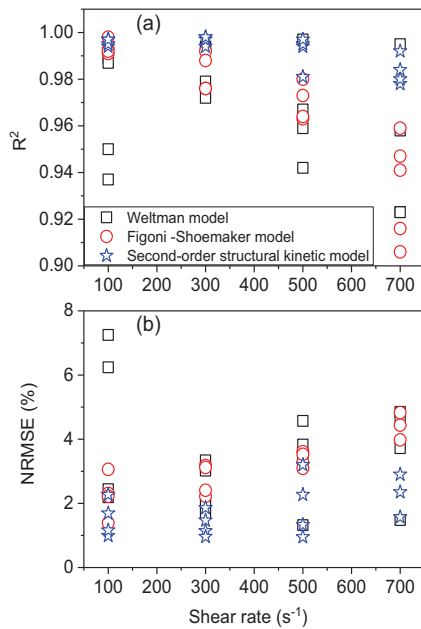


**Figure 7.** Rheological parameters of MPCM suspensions as the function of shear rates and concentration obtained by the Second-order structural kinetic model (Equation 3) at  $20^\circ\text{C}$ , (a) Equilibrium viscosity  $\eta_e$ , (b)  $[\eta_0/\eta_e]$  and (c)  $k_s$ .

increased. Breakdown of flocculates/agglomerates and orientation or deformation of suspended solid structures during the shearing process can cause a structural breakdown of suspensions.<sup>[16,20]</sup> When shear forces are applied to the suspensions, the agglomerates can be broken apart by hydrodynamic shear stresses (deflocculating effect), and structures can be built up by the increased amount of collisions induced by the shear (flocculating effect).<sup>[21]</sup> A balance of these two effects governs the breakdown process. At low shear rates, the forces are not large enough to break apart the agglomerated structures, and the flocculating effect dominates. At high shear rates the deflocculating effect is strong while the re-association rate is expected to be low, resulting in a significant viscosity decrease<sup>[19,22]</sup> As can be seen from Figure 3 and Figure 6, the agglomerated microcapsules breaks down to smaller sizes during the initial stage of applied shear forces. The deflocculating effect is dominant in this stage, resulting in a sharp decrease in the shear stress and the viscosity. At longer times, the agglomerates become smaller, and the shear forces do not have any significant effect on the overall size of the agglomerates.

The distance between the microcapsules becomes shorter at higher concentrations, leading to an increased interaction between the particles and the formation of larger agglomerates. When a shear force is applied, these large agglomerates are continuously sheared to smaller pieces. According to Fignon-Shoemaker,<sup>[10]</sup> the extent of the attractive forces between agglomerates depend on the size of the agglomerates. They predicted that the breakdown rate of large agglomerates is higher than for the smaller ones.

If the deflocculation rate is higher than the flocculation rate, a decrease of the viscosity and shear stress occurs over



**Figure 8.** The comparison of (a)  $R^2$  and (b) NRMSE for the Weltman model, the Fighi-Shoemaker model and the second-order structural kinetic model.

time (the Weltman model). In addition, the Fighi-Shoemaker model and second-order structural kinetic model predicts that an equilibrium is reached after some time, where both of these effects are equal in magnitude. In order to examine which of the three models provide the best fit to the time-dependent rheological behaviour of the MPCM suspensions, the determination coefficient  $R^2$  (Figure 8a) and normalized root mean squared error (NRMSE) (Figure 8b) is compared for the three models. The coefficient of determination  $R^2$  ( $0 \leq R^2 \leq 1$ ) is an evaluation of how well the fitted curve represents the experimental data.  $R^2 = 1$  would indicate that the fitted line fits the experimental data perfectly. As can be seen from Figure 8a,  $R^2$  varies in the range of 0.92–1 for the Weltman model, 0.91–1 for the Fighi-Shoemaker model, and 0.99–1 for the second-order structural kinetic models provided the best fit to the data. However,  $R^2$  values may not always be a true indicator of how well the model fits the data, particularly when a large number of data points are analyzed.<sup>[22]</sup> The NRMSE values can therefore offer a better picture. NRMSE is frequently used to normalize the differences between fitted data and experimental data. It is employed to compare different models which do not utilize the same scales. A lower value of NRMSE indicates less residual variance and a model that fits the data better.

Figure 8b shows that the NRMSE values are 1.32–7.25 for the Weltman model, 1.39–9.84 for the Fighi-Shoemaker, and 0.95–3.2 for the second-order structural kinetic model again illustrates that this model can be considered as the best model for characterizing these systems. This is also in agreement with a visual inspection of the fitted lines in Figure 3 and Figure 6, where the second-order structural kinetic model (Figure 6) follows the

experimental data better than the Weltman and Fighi-Shoemaker models (Figure 3).

## Conclusions

The influence of steady shear on the time-dependent structural breakdown of microencapsulated phase change materials (MPCM) suspensions were analysed by the Weltman model, the Fighi-Shoemaker model and the second-order structural kinetic model. The second-order structural kinetic model was found to exhibit the best correlation with the experimental data. The MPCM form agglomerates, which are disrupted by shear forces. The breakdown of the agglomerated structures was most pronounced at high shear rates where the microcapsules are subjected to stronger disruptive forces. In addition, more and probably larger agglomerates are present at higher concentrations, which causes a stronger breakdown of the agglomerated structures when the concentration is raised.

## Acknowledgement

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