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ORIGINAL CONTRIBUTION

Stimulated aggregation, rotation, and deformation of magnetite-filled microcapsules in external magnetic fields

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Abstract Microcapsules are nowadays applied in a wide range of products including food, pharmaceuticals, and cosmetic formulations. Because of their pronounced viscoelastic properties, polymer microcapsules are able to undergo mechanical deformations when they are stimulated by external signals. The elongation of capsule membranes, up to bursting processes, is important for the controlled release of active ingredients under well-defined conditions. Their application as quick and reliable control release systems affords a detailed knowledge of their rheological and mechanical properties. The aims of our work were to prepare new types of magnetic switchable microcapsules and to investigate their deformation behavior in externally imposed magnetic fields. The magnetic sensitivity was achieved by encapsulating magnetic magnetite (Fe_3O_4) particles within a polyorganosiloxane capsule. We investigated the dynamic response of those capsule suspensions and single capsules to external magnetic forces where aggregation processes, rotational movements, and field-induced deformations in static or rotating fields were observed.

Keywords Microcapsules · Polyorganosiloxanes · Interfacial polymerization · Magnetite particles · Field-induced deformation

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Introduction

Because of their controlled release properties, microcapsules are often synthesized for technical, cosmetic, medical, and pharmaceutical applications [1–11]. The semipermeable polymer membrane often controls the exchange processes between the external medium and the inner liquid-like core [1–11]. Transport procedures might be driven by osmotic forces or diffusion processes [11]. The membrane acts as a semipermeable barrier, just allowing a limited flow of the inner compounds towards the outer phase. It is, therefore, possible to release drugs in a controlled and defined manner over long periods [12, 13].

Mechanical deformations induced by stirring actions, gravity effects, reaction heats and diffusion processes often induce membrane oscillations [14, 15]. The ability of electric or magnetic field-sensitive capsules and membranes to undergo rapid changes in shape and size can be used to mimic artificial muscular contractions or to develop small reactors and nanomachines with tailor-made properties [16, 17]. Because of their simple synthesis and defined structures, microcapsules can also serve as model systems for the evaluation of mechanical properties of biological cells. Three different types of deformation can, at least, be distinguished: purely elastic, viscoelastic, and viscous membrane response [18]. Each of these processes can cause characteristic time-dependent deformations after the application of external fields like shear flow, magnetic, or electric forces.

Different approaches to synthesize polymer micro- and nanocapsules have been successfully undertaken, and their use in drug delivery is often desirable [1–11]. Special research interests focus on polyamide [14, 15, 19–21] and polysiloxane microcapsules [14, 15, 22–25]. Hydrogel capsules of alginate, gelatin [26–28], and polyvinylalcohol

[28] are already commonly employed in food technology and pharmacy. Hollow microspheres of polyelectrolytes [13, 29–31] prepared by the layer-by-layer technique [32, 33] are widely developed and extensively characterized for special applications.

A simple emulsion technique can be used to synthesize polyorganosiloxane microcapsules [14, 15, 22–25]. The viscoelastic shell around these particles can be formed by the interfacial condensation of long chain alkyltrichlorosilanes, such as octadecyltrichlorosilane (OTS) in a water/oil two-phase system. The interfacial network formation resulted from a two-step reaction: first hydrolysis of OTS and then subsequent condensation of the generated octadecyltrichlorosilicol [15, 22, 34, 35]. The polymerization reaction only occurs at the interface between oil and water. During this reaction, the surfactants are transformed from a sol state to a two-dimensional gel state. The cross-linking reaction leads to the formation of a stable, two-dimensional polyalkylsiloxane network in the vicinity of the oil–water interface [15, 22, 34–38]. The ultra-thin network exhibits pronounced rubber-elastic properties [15]. The network is semipermeable, but depending on the silane concentration the pore sizes vary [12, 15]. These pores serve as transport channels when trapped-reactive agents have to be released under deformation forces [12, 15]. Mark [39] reported on the incorporation of molecules like elastomers, proteins, claylike-layered, and magnetite particles into the two-dimensional polysiloxane network by an in situ process during the gelification reaction. These filler particles can modify the mechanical, electric, or magnetic properties of the ultra-thin membranes. These tailored capsules can easily be stimulated by external signals.

In a series of measurements, we synthesized and characterized polymer capsules whose polysiloxane networks and inner liquid cores contained magnetic particles of magnetite (Fe_3O_4). Magnetite particles in a suspension are sensitive to magnetic fields already at rather low field intensities between 10 and 100 mT [40]. Two different, basic interactions are often distinguished: *particle–particle interactions* and the *field–particle interactions* [41, 42]. In an almost *uniform magnetic field*, the magnetite particles tend to form permanent magnetic dipoles which are oriented parallel to the direction of the applied field. Because of the resulting attractive dipolar *particle–particle interactions*, the particles form rod- or chain-like structures which are geared to the lines of magnetic flux. The imposed field as well as the mutual particle interactions are simultaneously present and establish the moment-inducing field [40–45].

In a *non-uniform magnetic field*, the magnetite particles experience a magnetophoretic force along the gradient of the magnetic field intensity. The *field–particle interaction* becomes dominant and the particles are attracted to the region of higher field intensity because their permeability usually exceeds the permeability of the fluid medium (positive magnetophoresis). When the magnetite particles are bound to a polymer membrane, the magnetophoretic forces are transmitted to the surrounding polymer structure, and this can induce macroscopic membrane deformations or motions [41, 42].

Experimental: chemicals and methods

Chemicals

OTS was purchased from ABCR, stored under argon, and used without further purification (95% purity). *n*-Dodecane p.a. was obtained from Fluka, purified by means of a silica gel column, and dried over anhydrous sodium sulfate. Water was obtained from an ultra-pure water system (Seralpur PRO 90 CN, USF Seral).

The iron oxide particles were prepared in water by means of a chemical precipitation process [43, 44]. Solutions of 3.0 g of $FeCl_3{\cdot}4H_2O$ in 12.5 mL of water and 6.0 g of $FeCl_2{\cdot}6H_2O$ in 12.5 mL of water were combined. Afterwards, 12.5 mL of ammonium hydroxide (14.8 M) was added under vigorous stirring. The resulting black precipitate of magnetite settled rapidly by placing a magnet below the beaker, and then the supernatant solution was decanted. Afterwards, the solid precipitate was resuspended in 25.0 mL of 0.75 M ammonium hydroxide. The solid particles were then isolated using magnetic-induced separation again, decanted, and washed three times with pure water. The magnetic particles were not stabilized, so they formed aggregates with typical lengths of several micrometers which precipitated, because of density differences, within a few minutes. Redispersion of the particles was induced by sonification shortly before the magnetite-filled capsules were prepared.

Preparation and characterization of microcapsule suspensions

The formation of the capsule membrane was induced by the interfacial polycondensation of OTS at the interface between *n*-dodecane and water. For this purpose, 10 mL of ultra-pure water was emulsified in 30 mL of the OTS-containing dodecane phase by stirring with a magnetic stirrer with maximum speed (1.100 rpm). The concentration of OTS in dodecane differed in a range of 0.1 to 1.0 mmol/L. The polycondensation starts automatically when both phases get in contact. The reaction time was 7 min. After precipitation of the capsules, the organic phase (with the rest of the monomer) was exchanged by pure organic liquid for three times. All procedures have been carried out at room temperature (21°C).

To prepare capsules which were filled with magnetic material, we used the same procedure described above by replacing the pure water by the magnetite suspension. The concentrations of the magnetic particles differed in a range from 0.125 to 1.25 g/L.

To characterize the microcapsule suspensions and the magnetic particles inside the capsules, we mainly used microscopic methods. We used an Olympus BX 50 Microscope and placed a few drops of the capsule suspension with a small amount of the organic liquid (dodecane) onto a glass slide with a swilley. A representative number of micrographs were taken and used for the manual determination of the capsule size distribution.

To investigate the behavior of the magnetite-filled capsules in magnetic fields, we used static and dynamic (rotating) magnetic fields with intensities about 10 mT. To create a static magnetic field, we used a permanent magnet. The strength and the orientation of the magnetic field depend on the distance between the sample and the magnet as well as on the orientation of the magnets (horizontal or vertical) in relation to the sample. A rotational magnetic field was created by a magnetic stirrer, and to determine its influence on the sample, a few drops of the capsule suspension were given on a glass slide onto the stirrer. The effective magnetic fields are measured by a Hall probe.

Preparation and deformation of sessile drops and capsules

The field-induced deformation of magnetite-filled capsules can be well characterized by monitoring a single capsule. To prepare a single sessile capsule, we filled a glass cell with the organic phase which contained the monomer (OTS). The concentration of the monomer in the organic liquid was 0.1 mmol/L. Then, we placed one single drop of the water phase, which contained the magnetic material in different concentrations, into the organic phase and the polymerization process started directly. The deformation of a capsule was induced by an electromagnet stored above the capsule. To investigate the magnetic behavior of the prepared sessile capsule, we used a modified optical drop shape analysis instrument (OCA20 from DataPhysics). For details of the modification, see the Supplementary material. The instrument calculated the interfacial tension γ by means of the shape of the drop respectively capsule using the Laplace–Young formula.

It is important to mention that the calculation is not exactly valid for capsules because the gel point of the polymer membrane is achieved and then the elasticity of the solid-like membrane determines the capsule shape. At this point, we just use the detected interfacial tension values to determine the degree of capsule deformation and called it apparent interfacial tension or marked it with an asterisk [46].

Results and discussion

Shape, size, and structure of the polyorganosiloxane microcapsules in dependence on the magnetite concentration

Shape and size

In a series of experiments, we synthesized microcapsules containing various amounts of magnetite particles (0.125 to 0.5 g/L). The shape, size, and structure of these capsules could be obtained from light optical micrographs. Typical results of these investigations are summarized in Fig. 1.

The micrographs did not show considerable changes in size and shape of the microcapsules when the magnetite concentration was increased. It is evident, however, that at elevated magnetite concentrations, rod-shape aggregates were formed in the core regimes of the microcapsules.

The histograms in Fig. 1 describe the size distribution of the polymer microcapsules when the magnetite concentration is elevated. Measured values for the average diameter $\overline{d}$, the standard deviation $\sigma(d)$, and the number of measured capsules N are summarized in Table 1.

The histograms and the mean size values do not reveal a significant influence of the magnetite concentration, neither

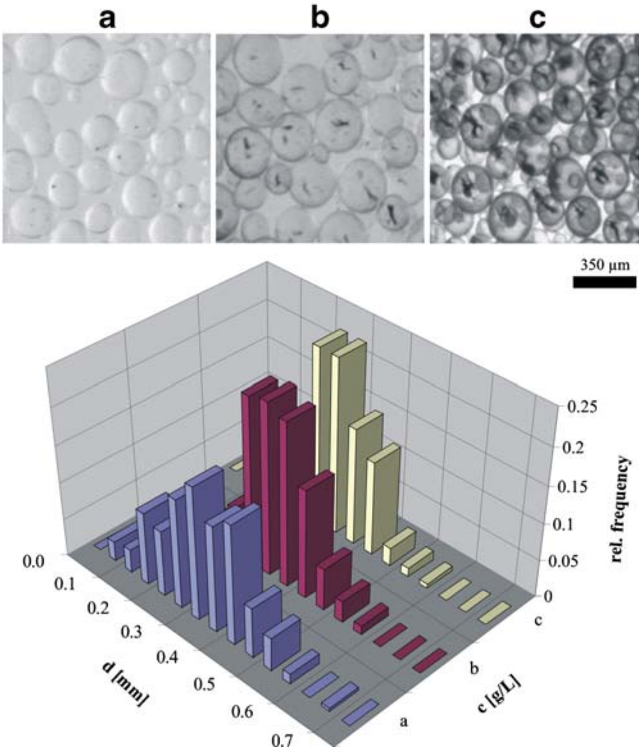

Fig. 1 Light optical micrographs and size distribution (relative frequency) of polyorganosiloxane capsules. Outer phase: *n*-dodecane (volume fraction 75%). The reacting monomer was dissolved in the oil phase (c(OTS)=1 mmol/L). Inner phase: **a** c(Fe_3O_4)=0.125 g/L; **b** c(Fe_3O_4)=0.25 g/L; **c** c(Fe_3O_4)=0.5 g/L in water

on the capsule size nor on its distribution. The mean capsule diameter of the investigated systems was of the order of 330 μm.

In particular, compared to former results of the synthesis of polyorganosiloxane capsules without magnetic particles [46], it could be shown that the capsule sizes are not affected by the presence of magnetite particles in the water phase. This gives us the opportunity to prepare capsules filled with magnetic materials in an easy way.

Structure

As a result of the surface polymerization, a stable, network-like membrane was formed at the interface between *n*-dodecane and water [15, 34]. Typical results obtained from light optical microscopy are documented in Fig. 2.

It is obvious that tiny magnetite particles (black regions) were fixed on the surface of the capsule membrane. Close inspection reveals that the magnetic particles were less homogeneously distributed onto the membrane. The particles are big enough for optical investigations, so we determined their particle sizes by analyzing their light optical micrographs. Most of the magnetic particles were smaller than 5 μm but we also detected some particles with sizes of about 30 μm. During the formation and precipitation process, magnetite particles of various sizes are generated and they tend to form aggregates. These aggregates become bigger during the sedimentation process. It is not possible to distinguish between single particles and aggregates, but it is obvious that the formation of aggregates was not induced by a magnetic field because the optical light micrographs were taken before a magnetic field was applied.

The light optical micrographs suggest that a part of the particles is fixed on the surface. This could happen by a simple adsorption process or a chemical reaction between the oxide surfaces of the magnetite particles and the polymer (OTS) surface. As the magnetite was almost homogeneously distributed in the water phase, we assume that a part of the iron oxide nanoparticles was already in the vicinity of the oil–water interface when the surface polymerization started and could therefore be fixed to the membrane during the two-dimensional gelation process.

When the magnetite concentration was increased, the optical observation of the magnetic particles became more difficult because of the large contrast contribution of the magnetite containing inner liquid. We observed, however, that the amount of membrane-adsorbed particles increased at elevated magnetite concentration in the aqueous phase.

Table 1 Mean values of capsule size $\overline{d}$, standard deviation $\sigma(d)$, and number of measured particles *N* for three different magnetite concentrations

c, g/L	*N* (capsules)	$\overline{d}$, μm	$\sigma(d)$, μm
0.125	225	327	113
0.25	178	345	81
0.5	165	314	87

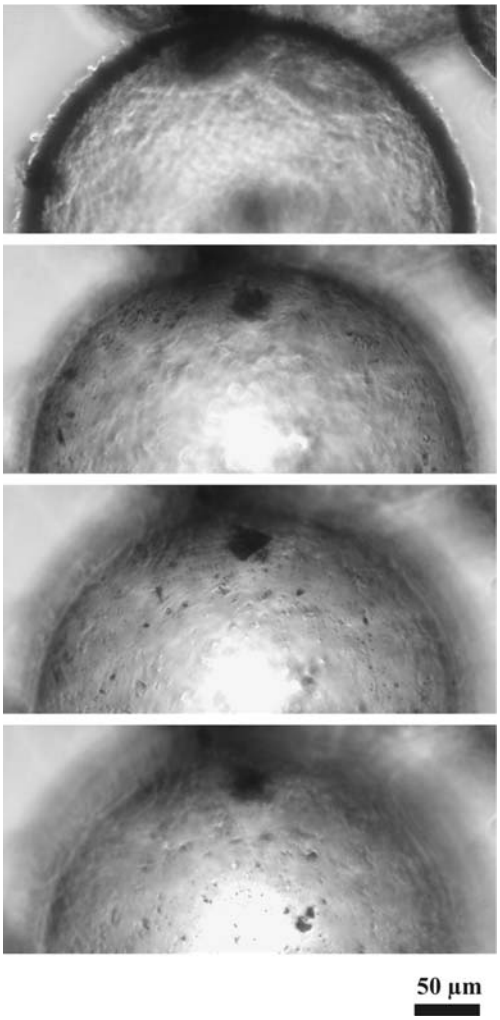

Fig. 2 Microscopic images of a microcapsule membrane which was investigated at different focus layers. Outer phase: c(OTS)=0.5 mmol/L in *n*-dodecane. Inner phase: c(Fe_3O_4)=0.125 g/L in water

Orientation of the magnetic particles in the inner liquid microcapsule cores in a static magnetic field

Without the influence of external magnetic fields, the iron oxide particles are randomly distributed, and they tend to sedimentate within the capsule cores because of the action of gravitation forces. In static magnetic fields, however, the magnetite particles can be reoriented along the lines of magnetic flux. This leads to the formation of elongated, rod-shaped aggregates with typical diameters of about 20 μm and lengths of about 400 μm. In the first image of Fig. 3, the magnetic flux is oriented in the direction of the arrow (*y*-axis). It is easy, to see, that the magnetite needles in the core of the capsule are also oriented in this direction. When the magnetic field is turned by a certain angle, the particle aggregates spin around always following the movement of the external magnetic field.

From the light optical micrographs, it is obvious that the transparency of the magnetite-filled microcapsule suspension can be influenced just by switching the orientation of the magnetic field vector. The magnetite particles are all colored black. The detected intensity of light passing through the suspension of magnetite-filled microcapsules depends strongly on the orientation of the iron oxide aggregates. It turns out that the light intensity increases when the magnetite chains are oriented parallel to the direction of incident light (see second image in Fig. 3). If the long axis of the aggregates is oriented perpendicular to the direction of incident light, the transparency is considerably reduced as shown in the first image of Fig. 3.

Orientation of the magnetic particles in the inner liquid microcapsule cores in a rotating magnetic field

Figure 4 presents the time-resolved orientation and movement of magnetite aggregates within the microcapsules when a rotating magnetic field is applied. To elucidate the field-dependent behavior of the magnetite particles, the concentration of microcapsules was increased. Because of the close packing, the capsules could not move and their positions were fixed on the glass slide. During the rotation of the magnetic field, the magnetite particles formed needle-like aggregates which oriented towards the poles of the magnetic field and followed their rotational movement.

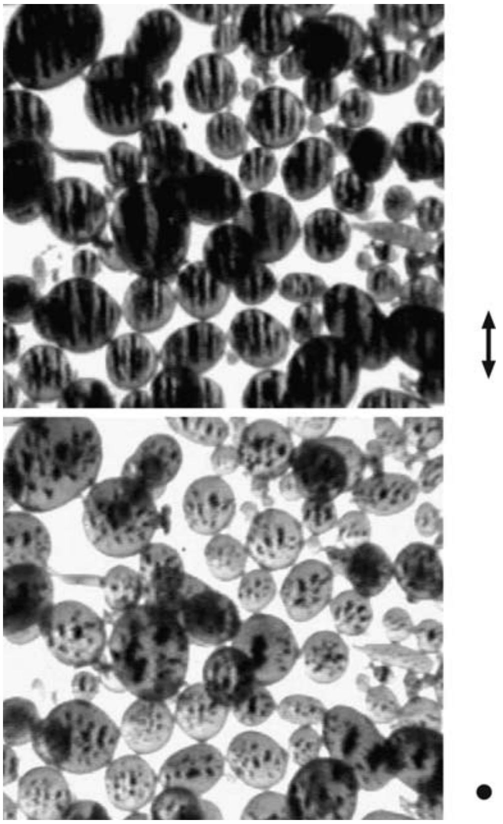

Fig. 3 Magnetic field induced orientation of magnetite particles within the liquid core of microcapsules (c(Fe_3O_4)=0.5 g/L, c(OTS)= 1.0 mmol/L). Magnetic field intensity: 6–8 mT. *First image*: The magnetic field vector is vertically directed within the paper plane (*arrow*). *Second image*: The magnetic field vector is directed perpendicular to the paper plane

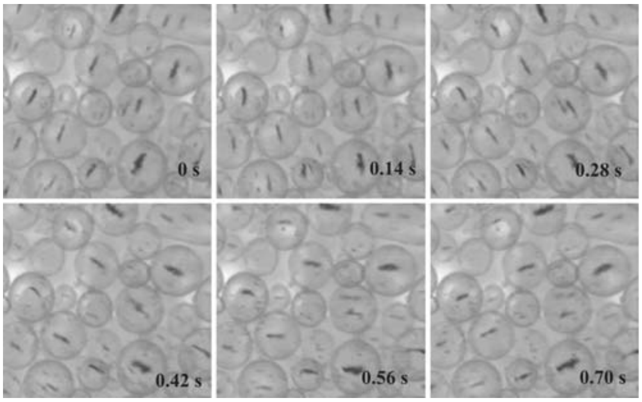

Fig. 4 Time-resolved orientation of magnetite particles within the microcapsules in an external rotating magnetic field. The orientation of the magnetic field vector changed with a velocity of about 30 rpm (rotation anticlockwise). Outer phase: c(OTS)=1 mmol/L in *n*-dodecane. Inner phase: c(Fe_3O_4)=0.25 g/L in water. Magnetic field intensity: 6–8 mT

The angular frequency of the magnetite aggregates coincided with the rotation movement of the external magnetic field. In a series of experiments, we could increase the rotational speed up to values of about 1.100 rotations per minute (rpm). At velocities above this threshold value, the particle aggregates were destroyed because of the action of centrifugal forces.

Orientation of the magnetite-filled microcapsules in a rotating magnetic field

A differing response of magnetic capsules was observed for more diluted capsule suspensions. The investigated capsules contained higher concentrations of magnetic particles inside the capsule core and onto the membrane. In this way, their mobility was enhanced and the external magnetic field was now strong enough to move the capsules. Typical results of these investigations are given in Fig. 5.

The light optical micrographs demonstrated that single capsules formed elongated chains of lengths up to 1 mm. These pearl-like aggregates were oriented along the lines of magnetic flux. Caused by the rotation of the external magnetic field, the aggregated chains also started to turn around in the same manner. It is interesting to note that single capsules sometimes were transferred from one chain to another during the rotation. This is exemplarily seen for one capsule in Fig. 5. It is evident that a single capsule (a)

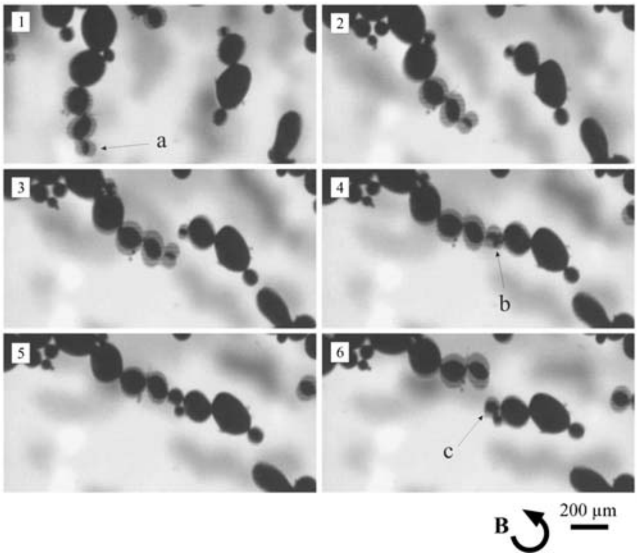

Fig. 5 Orientation of magnetite-filled polymer microcapsules observed in a rotating magnetic field (30 rpm); the difference between the images corresponds to time intervals of 0.14 s. The pictures should be viewed from numbers *1* to *6*. The *letters* indicate interesting points which are mentioned in the text. Outer phase: c(OTS)=1 mmol/L in dodecane. Inner phase: c(Fe3O4)=1.5 g/L in water. Magnetic field intensity: 6–8 mT

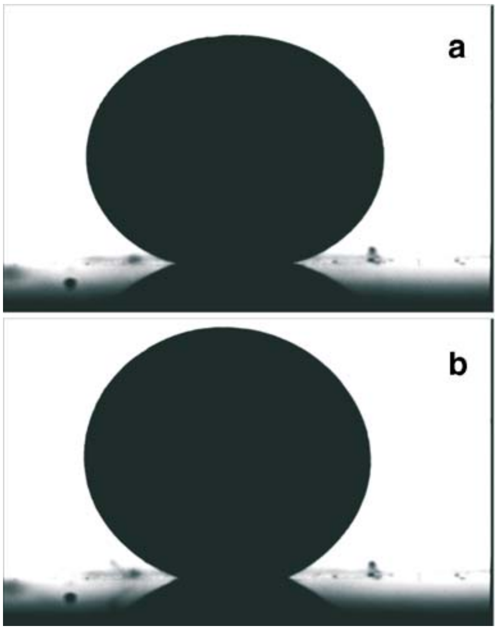

Fig. 6 Deformation of a sessile capsule filled with a magnetic particle suspension induced by a magnetic field. The magnet is located above the capsule. Outer phase: c(OTS)=0.1 mmol/L in dodecane. Inner phase: c(Fe_3O_4)=1.5 g/L in water. **a** Magnetic field is out; **b** magnetic field is on. Magnetic field intensity=7–9 mT

was transferred from one chain via a contact point (b) to another chain (c) during the rotation. The capsule transfer might be induced by spatial and temporal fluctuations of the magnetic field intensity. Fluctuations arise when the external magnet and the capsule chains do not move synchronically to each other. Because of friction forces, the rotation of the capsule chains is slower than the rotational movement of the permanent magnet. So, the magnetic forces on the capsules locally varied.

Microcapsule deformation in a static magnetic field

The inclusion of a large amount of the magnetite particles within the liquid inner capsule core as well as the adsorption of another large amount of the magnetite particles onto the capsule membrane during membrane polymerization leads to an immobility of the particles. When a magnetic field is applied, the particles interact with the field and experience a force to move to a region of higher magnetic flux density. At this, they transfer this force directly to the membrane resulting in a deformation of the capsule visible as an elongation of the capsule. Figure 6 shows the vertical elongation of a sessile magnetite-filled capsule in an inhomogeneous magnetic field. The deformation can be clearly identified by the change of the capsule contour when the field is switched on.

As already explained in the experimental part, the deformation behavior of a single sessile capsule could be well characterized by monitoring its interfacial tension as a function of the reaction time in an alternating magnetic field. Figure 7 shows the course of the interfacial tension of two capsules with differing magnetite concentrations.

The capsule deformation can be deduced from the excursive rising of the interfacial tension when the magnetic field is switched on. In a magnetic field, the magnetite-filled capsule is pulled upwards, and the contact area decreases leading to the calculation of a higher interfacial tension. For the lower magnetite concentration (a) the effect of the magnetic field is nearly negligible. In

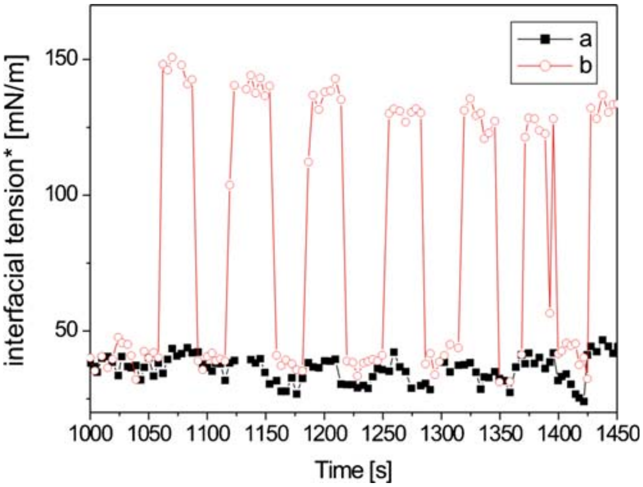

Fig. 7 Apparent interfacial tension versus time during the stepwise deformation of two different magnetite-filled capsules by an external alternating magnetic field. Outer phase: c(OTS)=0.1 mmol/L in dodecane. Inner phase: c(Fe_3O_4)=0.125 g/L (*a*) and 0.5 g/L (*b*) in water. Magnetic field intensity=7–9 mT

contrast, for the higher magnetite concentration (b) the influence of the alternating field is remarkably higher. As a result, we could prove that the degree of deformation is proportional to the amount of magnetite. When the magnetic field is switched off, the interfacial tension drops to its initial value meaning that the capsule returns to its initial shape pointing out the reversibility of the deformation. This means that the elastic properties of the membrane outweigh its viscous properties. In addition, our results showed that the degree of deformation also depends on the degree of polymerization of the polysiloxane network. The higher the monomer concentration is, the higher the degree of network polymerization is, and the lower the degree of deformation gets.

Conclusion

The interfacial polymerization of reactive silane monomers could be used to synthesize ultra-thin polymer microcapsules on the basis of a simple emulsion technique. Micrographs of the size distribution showed that the incorporation of magnetite particles into the polymer network did not affect the interfacial polymerization reaction in a significant way. Moreover, even the capsule size distribution was not affected by the presence of magnetic particles. The encapsulation of magnetite particles within the capsules led to a high magnetic field sensitivity when an external magnetic field was applied. While the magnetite particles in the capsule cores oriented along the lines of magnetic flux, the polymer capsules also formed straight chain-like structures in a uniform magnetic field. In a series of experiments, we could align and rotate the particle aggregates in a controlled manner just by applying external magnetic forces. Externally induced magnetic tensile forces were, however, transmitted from the magnetite particles to the polymer network. This led to stimulated magnetic-induced deformations of the magnetite-filled capsules whereby a high dependence of the degree of deformation on the magnetite amount and the reversibility of the deformation process in the chosen concentration range were proven.

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