Effect of centrifugal acceleration on stability evaluation

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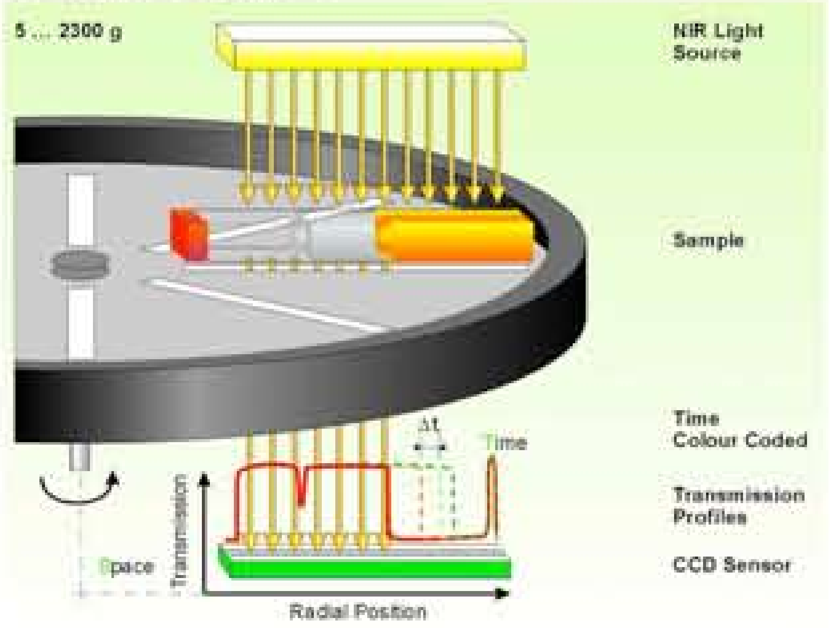
Scope

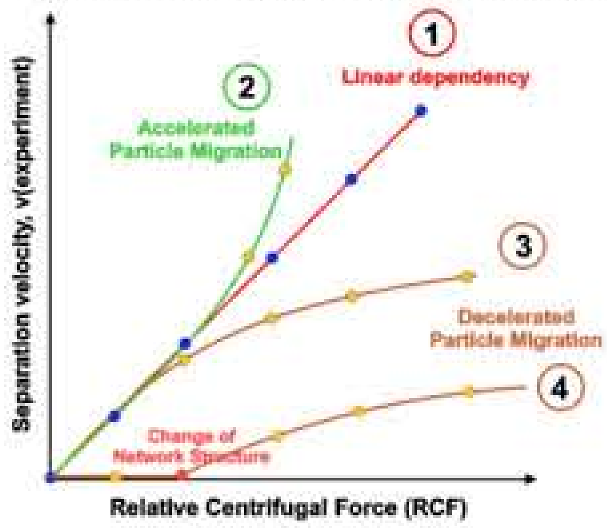
Multisample analytical centrifugation with high resolution photometric detection (STEP-Technology) is a useful tool for evaluation of the long term stability of dispersions. Sedimentation and creaming processes can be accelerated directly. Even more, changes in structural stability due to other destabilizing processes (coalescence, Ostwald ripening and flocculation) can be detected with high sensitivity and far more earlier than by visual observation at earth gravity, or by measurement of viscosity changes or particle size distributions.

On the other hand, the validity of results obtained in the centrifugal field for normal storage conditions is sometimes questioned. To this end a series of experiments was conducted on systems differing in polydispersity and interparticle forces (attractive/repulsive) measuring sedimentation or creaming at earth gravity and as function of centrifugal acceleration.

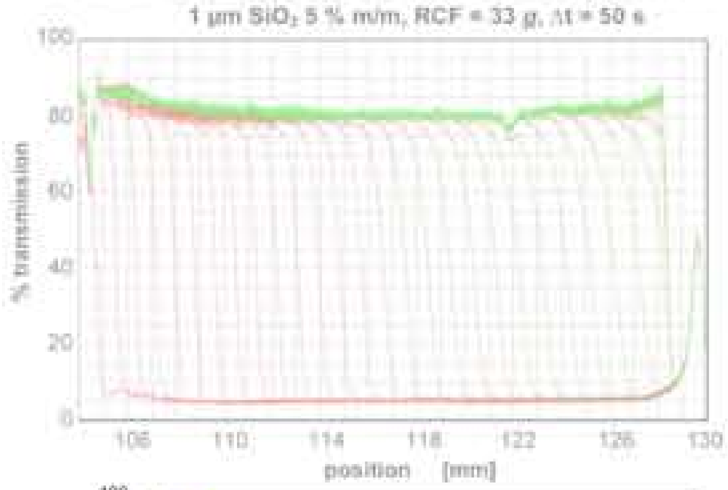


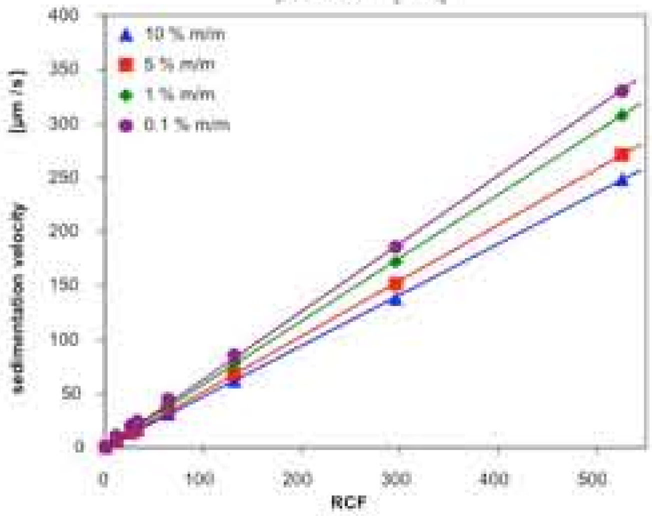
STEP-Technology - Local changes are detected during centrifugation. The distribution of transmission is recorded over the whole sample length at predefined time intervals.

Relation separation velocity acceleration



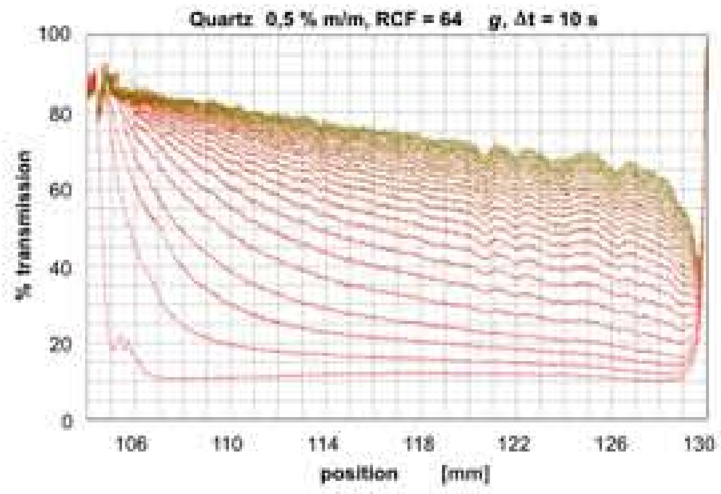
Stable monodisperse silica dispersions

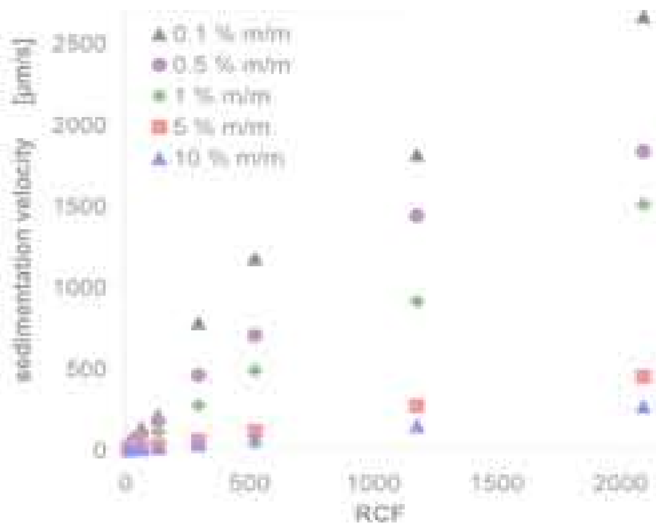




Monosized silica 1 µm, linear behaviour, with increasing solids concentration hindrance increases

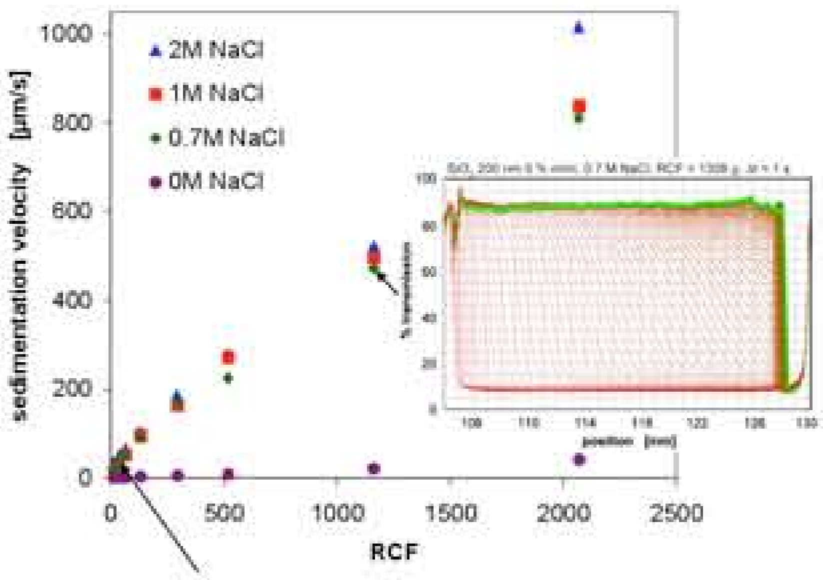
Stable polydisperse quartz dispersions

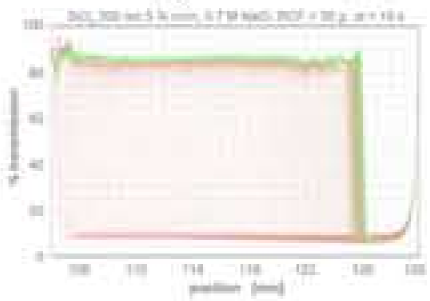




For the polydisperse quartz dispersion deviation from linearity was observed, which is due to a systematic error. At higher acceleration the fastest moving particles escape detection. Nevetheless behaviour at earth gravity can be safely deduced by nonlinear extrapolation approaching linearity at lower accelerations.

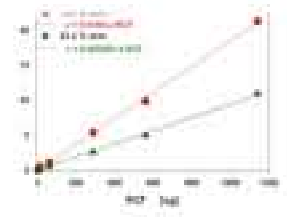
Flocculated silica dispersions

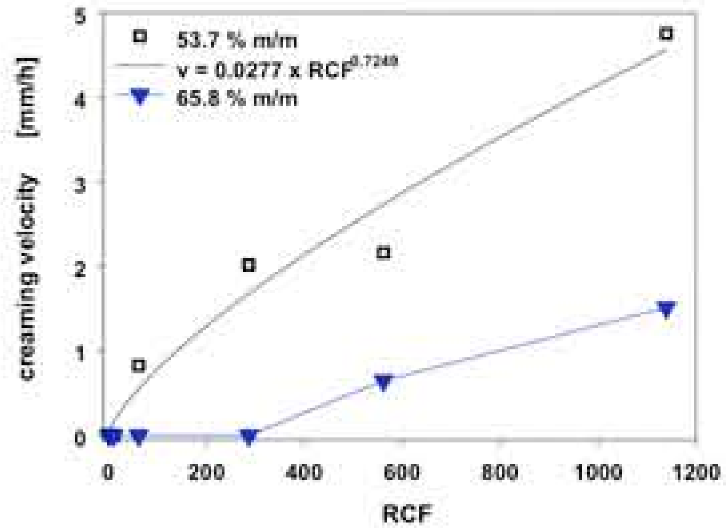




Monodisperse silica dispersions (5 % m/m, 200 nm) were destabilized by adding variable amounts of NaCl.
Velocities scale almost linearly except at high accelerations if at low degree of flocculation network breaks up – compare below.

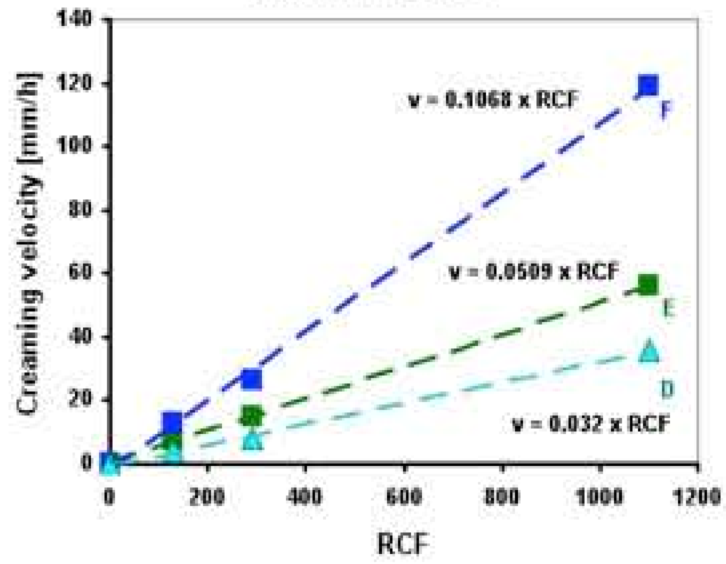
Paraffin oil in water emulsions





Creaming velocity scales linearly with RCF up to oil contents > 30 %. At high internal phase ratio a decelerated droplet migration was experienced, which can be fitted by a power law. However, when approaching the phase inversion point a yield point was observed, i.e. the emulsion is physically stable at normal gravity.

Cream liquers



Comparison of 3 cream liqueur samples. Samples differ in initial creaming rate. The creaming velocity scales linearly with RCF. This allows to calculate the physical shelf life at normal gravity.

Conclusion

- Different types of dispersions were evaluated regarding the separation velocity as function of the centrifugal acceleration applied.
- For stable monodisperse silica suspensions a linear dependence was observed with increasing hindrance at higher solids concentration.
- For stable polydisperse quartz suspensions a systematic deviation to lower velocities occured, however, behaviour at earth gravity can be extrapolated by nonlinear approach.
- For the flocculated silica the velocity scales almost linearily, except at high acceleration if at lower degree of flocculation the network breaks up.
- For oil-in-water emulsions linear behaviour changes to nonlinear when approaching the phase inversion point at higher oil content.
- **Shelf life at gravity may be experimentally determined by analytical centrifugation in hours instead of weeks or months (rapid testing).**

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