Evaporation Blocker for Cone-Plate Rheometry of Volatile Samples

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Abstract:
We report on the design and implementation of an evaporation blocker for cone-plate and plate-plate geometries. In addition to minimizing sample evaporation by trapping the saturated vapor inside a sample chamber, an important feature of the evaporation blocker is the suppression of solvent transport through condensation. Validation tests with DI-water, hydrogels, and colloidal suspensions demonstrate that the new accessory reduces solvent evaporation significantly more than commercially available environmental control chambers. Experiments were also performed to show that the evaporation blocker enables a variety of experiments on thermo-responsive complex fluids that were previously impossible, and provide new insight into the molecular organization of these materials. The evaporation blocker was specifically designed for an Anton Paar MCR rheometer, but the fundamental principles are widely applicable.

Zusammenfassung:
Wir berichten über das Design und den Einbau einer Verdampfungsbarriere für Kegel-Platte- und Platte-Platte-Geometrien. Eine wesentliche Eigenschaft zusätzlich zur Minimierung des Verdampfes der Probe durch Halten des übersättigten Dampfes innerhalb der Probenkammer ist die Unterdrückung des Lösungsmitteltransports durch Kondensation. Validierungstests mit DI-Wasser, Hydrogelen und kolloidalen Suspensionen zeigen, dass das neue Zubehör die Verdampfung des Lösungsmittels wesentlich mehr reduziert als kommerzielle Kontrollkammern für die Umgebung. Es wurden auch Experimente durchgeführt, um zu zeigen, dass die Verdampfungsbarriere eine Vielzahl von Experimenten an thermisch reagierenden komplexen Fluiden ermöglicht, die früher nicht durchführbar waren, und neue Erkenntnisse über die molekulare Organisation dieser Materialien liefert. Die Verdampfungsbarriere wurde speziell für ein Anton Paar MCR-Rheometer konstruiert, aber die Prinzipien sind in einem weiten Bereich anwendbar.

Résumé:
Nous reportons la conception et l'implémentation d'un stoppeur d'évaporation pour des géométries plan-plan et cône-plan. En plus de minimiser l'évaporation de l'échantillon en emprisonnant la vapeur saturée à l'intérieur du compartiment de l'échantillon, une caractéristique importante du stoppeur d'évaporation est la suppression de transport de solvant causé par la condensation. Des test de validation avec de l'eau dé-ionisée, des hydrogels, et des suspensions colloïdales, démontrent que ce nouvel accessoire réduit l'évaporation de solvant significativement mieux que les chambres de contrôle environnemental disponibles sur le marché. Des expériences ont également été réalisées afin de montrer que le stoppeur d'évaporation rend possible une variété d'expériences sur des fluides complexes thermo-sensibles qui étaient précédemment impossibles, et fournit un nouvel aperçu sur l'organisation moléculaire de ces matériaux. Le stoppeur d'évaporation a été spécifiquement conçu pour un rhéomètre Anton Paar MCR, mais ses principes fondamentaux sont largement applicables.

Key words: Evaporation, cone-plate rheometry, thermo-rheology, hydrogels, colloidal suspensions

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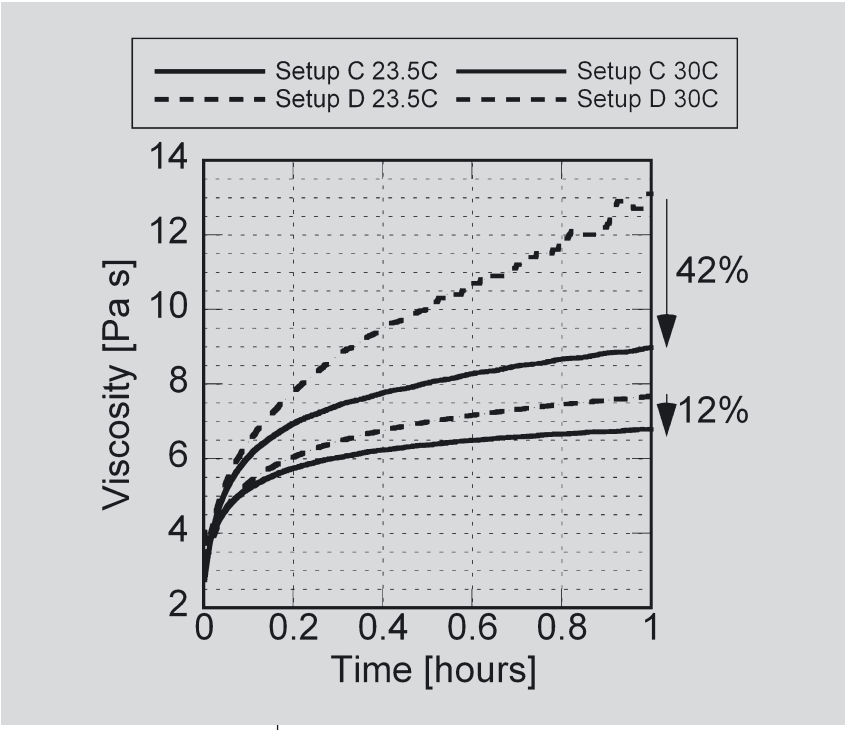

Figure 5: Transient viscosity of colloidal aqueous suspensions of precipitated calcium carbonate at 1 s^{-1} and different temperatures (Setup C and D).

To evaluate the effect of the immersion ring on sample evaporation, the experiments were repeated at a slightly elevated temperature, 30°C, only for the best performing configurations (Setup C and D). The results are shown in Fig. 5. After 1 hour, the measured viscosities without the immersion ring (Setup C) at room temperature, 23.5°C, and at 30°C were, respectively, 12 and 46% higher than with the immersion ring (Setup D). This suggests that the immersion ring dramatically prevents solvent evaporation even at temperatures that are only slightly higher than the ambient temperature. At higher temperatures, the effect of the immersion ring is expected to be even more dramatic.

4 CONCLUSION

We introduced a new rheometer accessory (evaporation blocker), which consists of an insulated sample chamber with temperature control elements (Peltier hood), vapor locks, immersion ring, and lower-plate inset with solvent trap. Through validation experiments with DI-water, we showed that this new accessory dramatically improves thermorheological measurements by minimizing solvent transport through evaporation and condensation.

The effectiveness of the individual parts of the system was investigated by using various combinations of the accessory parts. It was found that the biggest improvement in rheometer performance can be achieved by installing both the immersion ring and the solvent trap in the bottom plate to complement commonly used insulated chambers and vapor locks. We showed that the complete evaporation blocker set-up works very well for different cone diameters, 25 and 50 mm, and different complex fluids.

The accessory enabled experiments on concentrated colloidal suspensions and temperature-sensitive hydrogels that were impossible with commercially available evaporation protection (insulated chamber with Peltier elements and vapor lock). The new tool will be instrumental in providing new insight into the molecular organization of thermosensitive materials.

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