Novel Vacuum Compression Molding as a Tool for Sample Preparation in Rotational Melt Rheology

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Polymer processing equipment is designed based on the melt flow behavior and the throughput requirements. Rotational melt rheometry is widely applied to characterize the polymer melt flow behavior. Thereby, small samples are subjected to shear in an exactly defined geometry. Homogeneous samples are a prerequisite as the measured properties are averaged over the entire analyzed sample. The starting materials for an analysis are often powders or pellets, which need to be shaped to a specimen with a suitable preparation process. In such a preparation process, the sample material should not change its properties due to degradation. Conventional sample preparation approaches are for example compression molding with a heated platen-press or injection molding. However, both might be inapplicable for shear- and heat-sensitive materials as pharmaceutical substances.

This work introduces a novel vacuum compression molding (VCM) tool for the preparation of thermoplastic specimens. The VCM tool uses only standard laboratory equipment: a hot plate and a vacuum source. Unwanted adhesion between mold and sample is eliminated by applying PTFE coated separation foils, which fully enclose the sample and they can be easily peeled off from the sample after preparation. The sample compression is due to atmospheric pressure, when the tool is evacuated. In addition, this compact design enables short preparation times and low thermal loading.

The tool is applied in a rheological study for sample preparation of three pharmaceutical polymers. The preparation cycles were below 10 minutes for one sample. The samples were homogeneous and transparent, without voids or air inclusions. The subsequent rheological measurements showed high reproducibility as all relative standard deviations between repetitions were below 3%. The obtained rheological data were fitted via the Carreau-Yasuda model and time-temperature superposition was applied. The study demonstrates that sample preparation by VCM leads to highly reproducible measurement, even when heat sensitive materials are investigated.