MICROWAVE ASSISTED VACUUM DRYING AND ENDPOINT DETERMINATION USING MASS SPECTROMETRY

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ABSTRACT

Microwave drying technology has the potential for large production time and energy cost savings versus traditional vacuum drying methods for active pharmaceutical ingredient (API) applications. One of the most critical parameters in the drying process is the determination of the end point, usually at a specified moisture content. On-line mass spectrometry (MS) was used as a tool to monitor microwave vacuum drying operations on laboratory scale. On-line MS steady state response was shown to correlate with product dryness during laboratory microwave vacuum drying studies using several different solvents in calcium carbonate solids. The microwave vacuum drying profiles obtained with calcium carbonate showed a constant rate drying period followed by a short falling rate drying period. A method to estimate the loss-on-drying (LOD) and drying rate profiles based on MS data is demonstrated. The LOD method estimation is used to characterize the drying profiles for hygroscopic versus non-hygroscopic materials. The microwave drying rates were shown to have a linear correlation with solvent heat of vaporization and the solubility parameter; and depend on microwave power input and vacuum level. Process MS was also shown to correlate with other drying end point parameters, including rate of recovered solvent, product temperature, and microwave reflected power. The MS technique was applied to monitoring the drying endpoint of multiple solvent wet cakes. In comparison to conventional vacuum oven dryers, microwave drying rates were 20-100 times faster.