Organic Process Research & Development **2004**, *8,* 867–872

Microwave Assisted Vacuum Drying and Endpoint Determination Using Mass Spectrometry, Part II, On-Line Moisture Content Profiling

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Abstract:

Quantitative methods were developed to compute on-line conventional loss-on-drying (LOD) profiles from mass spectrometry (MS) measurements. Good agreement between calculated LOD values and actual LOD measurements were obtained for single and multiple solvent wet cakes. Characteristic drying curve profiles were determined for calcium carbonate, sand, cellulose, starch, and silica gel solids, and the corresponding critical moisture contents were estimated. Although this work was developed for microwave drying, the methods apply to conventional dryer monitoring as well.

1. Introduction

Moisture content profiles for dryer operations are often developed using off-line LOD measurements, which requires breaking vacuum at individual time points and sampling of the wet cake. On-line moisture content profiling for drying operations has been performed via near-IR (NIR) spectroscopy for both laboratory and pilot scale microwave vacuum dryers,[1] but there are inherent disadvantages of the NIR technique as it requires direct sample contact and cleaning before each run. The advantage of MS is that sampling from the vacuum line of the dryer is inherently more homogeneous than trying to directly sample the product, since the vapor is generated by the entire product in the dryer. While mass spectrometry has been utilized to demonstrate correlation between MS response and percentage of volatiles from a Guedu pilot scale vacuum dryer,[2] the moisture content profiles were determined off-line using a thermogravimetric analyzer (TGA). The objective of this work was to develop methods for monitoring the LOD during microwave drying operations based on vapor phase measurements via MS.

Previously we have demonstrated the correlation of mass spectrometry with LOD for constant and falling rate drying regimes in conventional vacuum ovens.[3] The present work extends these findings and demonstrates for the first time the utilization of vapor phase MS data to compute LOD in real time. We present two methods to convert vapor phase MS signal during drying to a real time LOD and drying rate value.

2. Experimental Section

The microwave dryer and setup is described in Part I of this work.[4] Various solids were utilized for microwave vacuum drying experiments: calcium carbonate powder (J. T. Baker); microcrystalline cellulose (Avicel PH-102); silica gel (J.T. Baker, 40 μm particle size, 60 Å pore diameter); starch, potato (Aldrich); and sand (J.T. Baker, purified). The experimental parameters for the microwave vacuum drying of the various solids included the following: 200 g of solids, 175 g of methanol, microwave power of 150 W, vacuum level of 100 mbar, and an oven air temperature of 35 °C. The additions for the multiple solvent wet cake run included 80 g of ethyl acetate and 80 g of hexane. The wet cake is prepared by adding the solvent to solids and then initially mixing in the microwave vessel before the microwave power is applied. Vacuum is initially applied to the wet cake approximately 30 s before the microwave power is initiated. Argon is introduced at a constant flowrate of 10 standard cubic centimeters per minute (sccm) and is used as a reference gas to enable the determination of the MS relative response profile. To minimize headspace effects, experiments were also run at a higher argon flow rate of 100 sccm.

For the drying of methanol in calcium carbonate, a gas standard mixture of methanol in argon was used, and the "concentration method" (section 2.1) was applied to calculate the LOD and drying rate profiles. For the remainder of the methanol runs, a gas standard mixture was not available, and the "area method" (section 2.2, 2.3) was used to calculate the LOD and drying rate profiles.

2.1. Method 1. Accurate concentration profiles can be determined in a mixture via mass spectrometry by correcting for the ionization probability differences between the components using a single-point calibration method, which assumes a linear calibration of the solvent concentration up to 100%. To do this, a standard gas mixture containing each component with known concentrations (mol %) must be utilized, noting that the measured response of a given species is only valid on the MS machine and specific tune that were used to produce the measurement.[5] The on-line methanol concentration, flowrate, drying rate, and LOD profiles can

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10.1021/op049853s CCC: $27.50 © 2004 American Chemical Society
Published on Web 10/13/2004

be determined for a stream consisting of methanol and the argon reference gas as follows:

Sensitivity factors (SF) for methanol (m/z 31) and argon (m/z 40) are calculated by dividing their corresponding MS response (amps) by their respective calibration gas mole fraction (X_{MeOH}, X_{Ar}), eq 1a and b. In this instance 1 mol % methanol in argon calibration gas was utilized.

$$\mathrm{SF}_{\mathrm{MeOH}} = \frac{m/z\ 31}{X_{\mathrm{MeOH}}} = \frac{7.3 \times 10^{-13}\ \mathrm{amps}}{0.01} = 7.3 \times 10^{-11}\ \mathrm{amps} \tag{1a}$$

$$\mathrm{SF}_{\mathrm{Ar}} = \frac{m/z\ 40}{X_{\mathrm{Ar}}} = \frac{1.7 \times 10^{-10}\ \mathrm{amps}}{0.99} = 17.2 \times 10^{-11}\ \mathrm{amps} \tag{1b}$$

The normalization factor for methanol ($\mathrm{NF}_{\mathrm{MeOH}}$) can be defined as the ratio of sensitivity factors for argon and methanol (eq 2).

$$\mathrm{NF}_{\mathrm{MeOH}} = \left(\frac{\mathrm{SF}_{\mathrm{Ar}}}{\mathrm{SF}_{\mathrm{MeOH}}}\right) = \frac{17.2 \times 10^{-11}\ \mathrm{amps}}{7.3 \times 10^{-11}\ \mathrm{amps}} = 2.35 \tag{2}$$

Once the normalization factor has been calculated, it can be applied to experimental MS data. Multiplying the normalization factor and the methanol MS response value results in the normalized response, m/z 31*(t), relative to argon (eq 3).

$$m/z\ 31^*(t) = (\mathrm{NF}_{\mathrm{MeOH}})\ (m/z\ 31(t)) = 2.35[m/z\ 31(t)] \tag{3}$$

Using eq 3, a corrected total ion signal (I_t*) can be calculated (eq 4).

$$I_t^* = m/z\ 31^*(t) + m/z\ 40(t) \tag{4}$$

The component vapor phase mole fraction profiles are calculated by dividing the corrected ion signal by the total corrected ion signal (I_t*), eq 5a and b.

$$X_{\mathrm{MeOH}} = \left(\frac{m/z\ 31^*(t)}{I_t^*}\right) = \left(\frac{m/z\ 31^*(t)}{m/z\ 31^*(t) + m/z\ 40(t)}\right) \tag{5a}$$

$$X_{\mathrm{Ar}} = \left(\frac{m/z\ 40(t)}{I_t^*}\right) = 1 - X_{\mathrm{MeOH}} \tag{5b}$$

The methanol vapor flowrate ($\mathrm{d}V_{\mathrm{MeOH}}/\mathrm{d}t$), with units of standard cubic centimeters per minute (sccm), is calculated by multiplying the flowrate of the argon carrier gas (set during the experiment) by the ratio of methanol to argon, eq 6.

$$\frac{\mathrm{d}V_{\mathrm{MeOH}}}{\mathrm{d}t}(sccm) = \frac{\mathrm{d}V_{\mathrm{Ar}}}{\mathrm{d}t}(sccm)\left(\frac{X_{\mathrm{MeOH}}}{X_{\mathrm{Ar}}}\right) \tag{6}$$

Once the flowrate has been determined, the methanol drying rate ($\mathrm{d}m_{\mathrm{MeOH}}/\mathrm{d}t$, mass/time) is calculated by simply multiplying by the molecular weight and applying the ideal gas conversion factor (1 mole per 22 400 scc), eq 7.

$$\frac{\mathrm{d}m_{\mathrm{MeOH}}}{\mathrm{d}t}(\mathrm{g/min}) = \left(\frac{\mathrm{d}V_{\mathrm{MeOH}}}{\mathrm{d}t}(\mathrm{sccm})\right)\left(\frac{\mathrm{mol}}{22\ 400\ \mathrm{scc}}\right)(\mathrm{MW}_{\mathrm{MeOH}}(\mathrm{g/mol})) \tag{7}$$

Likewise, the recovered liquid mass ($m_{\mathrm{L}}(t)$) profile is calculated by integrating the drying rate versus time, eq 8.

$$m_{\mathrm{L}}(t) = \int_0^t \frac{\mathrm{d}m_{\mathrm{MeOH}}}{\mathrm{d}t}\,\mathrm{d}t \tag{8}$$

The LOD(t) profile is calculated as percent moisture of the wet cake, using the initial solvent mass (m_{Lo}), recovered liquid ($m_{\mathrm{L}}(t)$), and mass of wet cake ($m_{\mathrm{T}}(t)$) values (eq 9).

$$\mathrm{LOD}(t) = \frac{m_{\mathrm{Lo}} - m_{\mathrm{L}}(t)}{m_{\mathrm{T}}(t)} \times 100\% \tag{9}$$

The above procedure may also be applied for sample streams with three or more components, assuming gas standards are available for each component to enable computation of sensitivity factors. The accuracy of the concentration method is dependent upon identifying and measuring all potential components in the sample line. To obtain real time LOD profiles, this method assumes that you know the initial solvent load and that the on-line software method can compute the integral shown in eq 8 and calculate eq 9 in real time.

2.2. Method 2. For cases where a gas standard mixture is not available, the following "area method" can be used to estimate solvent drying rates and LOD profiles. The "area method" is based on the principle that the cumulative area under the MS relative response profile is proportional to the total amount of liquid evaporated from the wet cake (exemplified in Figure 2a). By utilizing a constant reference gas flowrate, the relative MS response represents a *relative* concentration of the dryer vapors and a measure of the relative drying rate. If the initial amount of liquid (solvent) and solids content is known, the MS relative response profile can be normalized to determine the recovered liquid profile, LOD profile, and drying rate as a function of time. The procedure for these calculations, using methanol as solvent and argon (reference gas) as an example, is as follows:

Using a known quantity of methanol, the total MS response area (A_{T}) for the duration of the drying period can be calculated (eq 10). Note: t_{f} is estimated as the time at which the MS response returns to a baseline value. Likewise, the temporal cumulative area ($A(t)$) can be generated by taking the integral of the methanol MS relative response (m/z 31/40) curve (eq 11).

$$A_{\mathrm{T}} = \int_0^{t_{\mathrm{f}}} (m/z\ 31/40(t))\,\mathrm{d}t \tag{10}$$

$$A(t) = \int_0^{t} (m/z\ 31/40(t))\,\mathrm{d}t \tag{11}$$

The calculated recovered liquid profile ($m_{\mathrm{L}}(t)$) is determined by taking the ratio of the cumulative response area ($A(t)$) divided by the total response area (A_{T}) and multiplying this ratio by the initial solvent mass (m_{Lo}), eq 12. Applying this equation gives a normalized recovered liquid versus time profile based on the initial liquid mass.

$$m_L(t) = \left(\frac{A(t)}{A_T}\right) m_{Lo} \qquad (12)$$

The LOD (t) profile is calculated using eq 9.

The drying rate profile (dm_{MeOH}/dt) is calculated by taking the time derivative of the recovered liquid profile, eq 13.

$$\frac{dm_{\mathrm{MeOH}}}{dt}(\mathrm{g/min}) = \frac{d}{dt}(m_{\mathrm{L}}(t)) = \left(\frac{m_{\mathrm{Lo}}}{A_{\mathrm{T}}}\right) m/z\ 31/40(t) \qquad (13)$$

2.2. Method 3. In a manner similar to that of Method 1, a normalization factor for methanol ($\mathrm{NF_{MeOH}}$) can be determined based on the MS response area. Dividing eq 1a by 1b, the relative response ratio can be expressed in terms of $\mathrm{NF_{MeOH}}$ and the volumetric flow ratio of methanol (dV_{MeOH}/dt) to argon (dV_{Ar}/dt), eq 14.

$$\frac{m/z\ 31}{m/z\ 40} = \left(\frac{\mathrm{SF_{MeOH}}}{\mathrm{SF_{Ar}}}\right)\left(\frac{dV_{\mathrm{MeOH}}/dt}{dV_{\mathrm{Ar}}/dt}\right) = \left(\frac{1}{\mathrm{NF_{MeOH}}}\right)\left(\frac{dV_{\mathrm{MeOH}}/dt}{dV_{\mathrm{Ar}}/dt}\right) \qquad (14)$$

Integrating both sides of eq 14 from time zero to baseline (t_f), with dV_{Ar}/dt constant, eq 15 is obtained. Equation 15 can be rearranged to solve for $\mathrm{NF_{MeOH}}$, with the integral of dV_{MeOH}/dt (from 0 to t_f) equal to the total volume of methanol (V_{MeOH}), eq 16. The methanol recovered liquid, LOD, and drying rate can be calculated utilizing $\mathrm{NF_{MeOH}}$ and eqs 3−9.

$$\int_0^{t_f} (m/z\ 31/40(t))\ dt = \left(\frac{1}{\mathrm{NF_{MeOH}}}\right)\left(\frac{1}{dV_{\mathrm{Ar}}/dt}\right)\int_0^{t_f} (dV_{\mathrm{MeOH}}/dt)\ dt \qquad (15)$$

$$\mathrm{NF_{MeOH}} = \frac{\int_0^{t_f} (dV_{\mathrm{MeOH}}/dt)\ dt}{(dn_{\mathrm{Ar}}/dt)\int_0^{t_f} (m/z\ 31/40(t))\ dt} = \frac{V_{\mathrm{MeOH}}}{(dV_{\mathrm{Ar}}/dt)A_{\mathrm{T}}} \qquad (16)$$

This methodology can be extended to calculate LOD profiles for multiple solvent wet cakes. Figure 1 shows the relative response ratio profiles for ethyl acetate (*m/z* 45/40) and hexane (*m/z* 57/40) in calcium carbonate, with solvent additions of 80 g each, using an argon flowrate of 100 sccm. Utilizing eq 16, normalization factors were calculated for ethyl acetate ($\mathrm{NF_{EtOAc}} = 15.12$, eq 17) and hexane ($\mathrm{NF_{Hex}} = 7.87$, eq 18), using respective calculated relative response areas of 13.45 and 26.43 (Figure 1). The total gas-phase volume for hexane and ethyl acetate was calculated using the ideal gas law [$V = (nRT)/P$] with a solvent mass of 80 g. The LOD profile can be calculated utilizing $\mathrm{NF_{EtOAc}}$, $\mathrm{NF_{Hex}}$, and eqs 3−9.

$$\mathrm{NF_{EtOAc}} = \frac{\left(\frac{80\ \mathrm{g}}{88.11\ \mathrm{g/mol}}\right)(22\ 400\ \mathrm{scc/mol})}{(100\ \mathrm{sccm})(13.45\ \mathrm{min})} = 15.12 \qquad (17)$$

$$\mathrm{NF_{EtOAc}} = \frac{\left(\frac{80\ \mathrm{g}}{86.18\ \mathrm{g/mol}}\right)(24\ 200\ \mathrm{scc/mol})}{(100\ \mathrm{sccm})(26.43\ \mathrm{min})} = 7.87 \qquad (18)$$

An alternative "area method" may be applied based on the correlation between the total MS relative response area (eq 10) and the initial amount of solvent mass for multiple runs. Figure 2a shows the methanol MS relative response profiles for pure methanol microwave vacuum dryer runs of 50, 100, 150, and 200 g. Figure 2b shows the methanol MS relative response area versus methanol mass. The points on the graph were determined from the area under the methanol MS relative response (*m/z* 31/40) versus time curve for each mass (g) from Figure 2a. The magnitude of the MS relative response area is shown to be linearly dependent on the amount of mass evaporated as expected. The slope of the linear fit was equal to 24.36 [(*m/z* 31/40)*(min/g)] and provides a calibration factor that relates the MS relative response area to the solvent mass. The calculated recovered liquid ($m_{\mathrm{L}}(t)$) profile is determined by dividing the dryer run cumulative area ($A(t)$, eq 11) by the slope of the response area versus solvent mass plot, eq 19, alternate to eq 12. The LOD and drying rate profiles are then calculated using eqs 9 and 13, respectively. Note that the slope is dependent on the specific MS and calibration parameters. Caution must be utilized when applying this method to multiple dryer runs, since the MS response may drift/vary from day to day (or run to run).

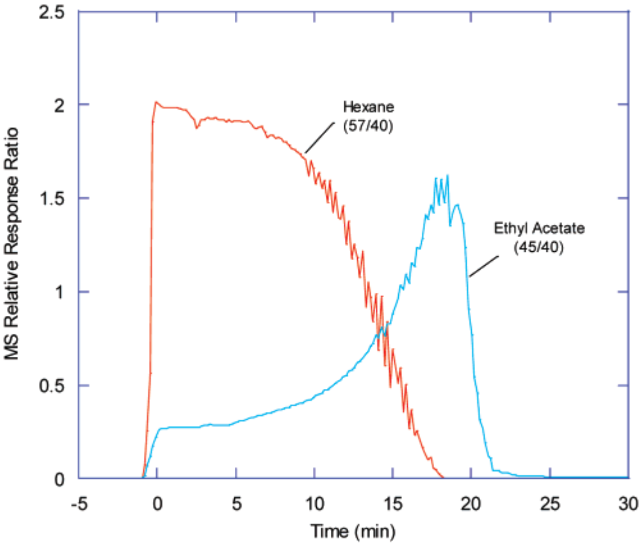

Figure 1. **MS relative response profiles for hexane (*m/z* 57/40) and ethyl acetate (*m/z* 45/40) in calcium carbonate (200 g), with solvent additions of 80 g each. The total MS relative response area (A_T) was 13.45 (*m/z* 57/40*min) and 36.43 (*m/z* 45/40*min) for ethyl acetate and hexane, respectively. The argon reference gas flowrate was 100 sccm.**

$$m_{\mathrm{L}}(t) = \frac{A(t)}{\mathrm{Slope(Area/Mass)} = 24.36} \qquad (19)$$

The advantage of the alternate "area method" approach is that the LOD profile can be calculated on-line, as long as the initial liquid and solids contents are known and the on-line software method can compute the integral in eq 11. Conversely, utilizing Method 2 (eqs 10−13), the LOD profile can only be estimated off-line (i.e., once the dryer run is complete). The disadvantage of the alternate "area method" approach is due to the upfront method development required, and the accuracy of the method is dependent on the stability of the MS calibration/response.

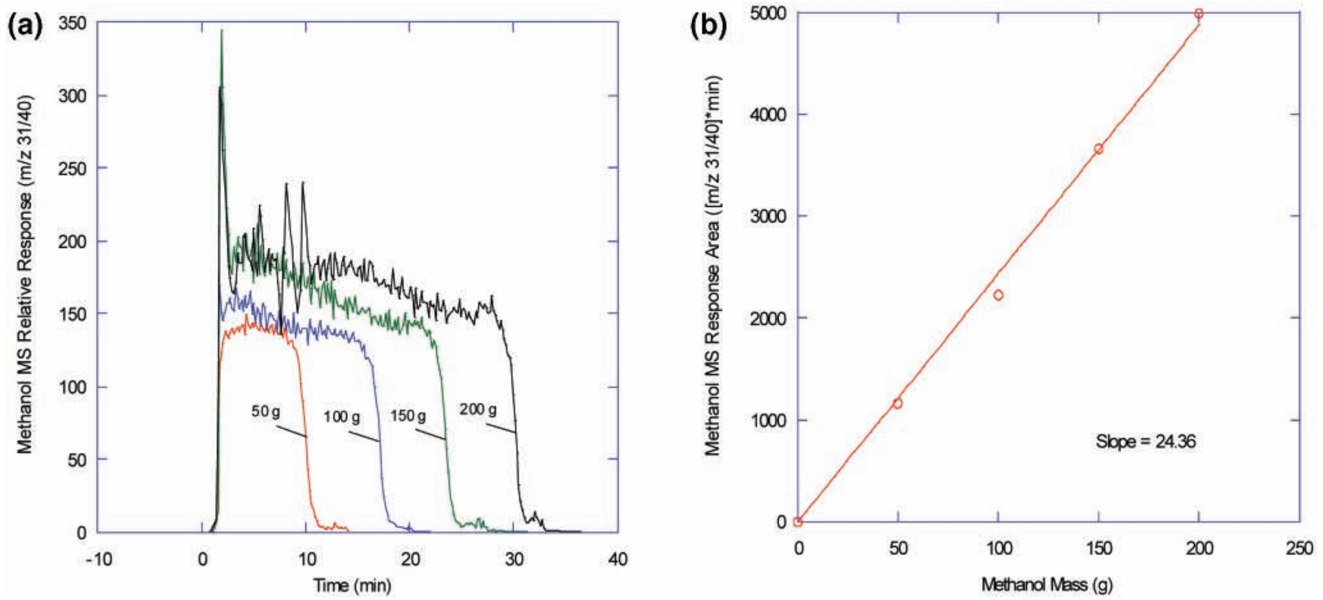

Figure 2. **(a) Methanol MS relative response for pure methanol microwave vacuum dryer runs of 50, 100, 150, and 200 g. (b) Methanol MS relative response area versus methanol mass, with a resulting slope equal to 24.36. The response area was determined using argon as a reference gas at 10 sccm.**

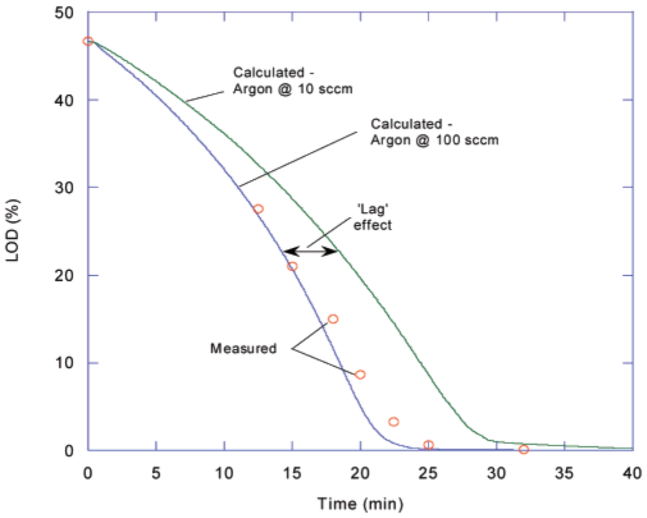

Figure 3. **Microwave vacuum drying profile of methanol (175 g) in calcium carbonate (200 g), showing measured LOD and calculated LOD based on Method 3. The calculated LOD utilizing an argon reference gas flow of 100 sccm correlates with the measured LOD.**

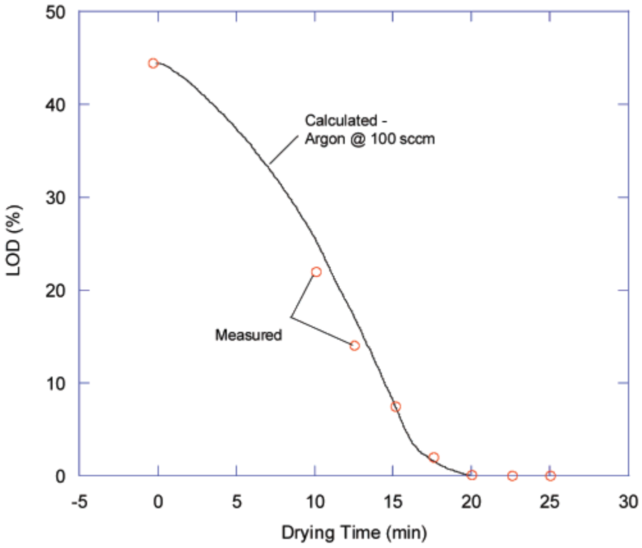

Figure 4. **Microwave vacuum drying profile of hexane (80 g) and ethyl acetate (80 g) in calcium carbonate (200 g), showing measured LOD and calculated LOD based on Method 3. The calculated LOD utilizing an argon reference gas flow of 100 sccm correlates with the measured LOD.**

3. Results and Discussion

3.1. LOD Profiling Using Method 3. 3.1.1. Single Solvent Wet Cakes. Using the MS relative response profile, the LOD versus time profile can be calculated. Figure 3 shows the measured and calculated (with argon flowrates of 10 and 100 sccm) LOD profiles of methanol in calcium carbonate, determined using Method 3. The LOD versus time profile is calculated using eq 9, and the recovered liquid (t) is determined using eq 19 and applying a slope value of 24.36. The calculated LOD profile for the 100 sccm argon flow rate was shown to correlate well with the measured (actual) LOD profile, whereas the calculated LOD profile for the lower argon flow (10 sccm) was shown to have a considerable lag and does not correlate as well with the measured LOD. The lag time is the time it takes for a change in the wet cake LOD to be detected in the vapor phase by the mass spectrometer downstream. Therefore, the larger the reference gas flow rate is, the closer the calculated LOD estimation will be to the actual values.

3.1.2. Multiple Solvent Wet Cakes. Using Method 3 the LOD profiles for multiple solvent wet cakes were calculated. Figure 4 shows the measured and calculated LOD profiles for hexane and ethyl acetate in calcium carbonate, using an argon flowrate of 100 sccm. The LOD versus time profile is calculated using eqs 3−9 with normalization factors of 7.87 and 15.12 for hexane and ethyl acetate, respectively, and was shown to correlate well with the measured (actual) LOD profile. Although the LOD decreases linearly with time for a majority of the drying period (5−18 min), Figure 1

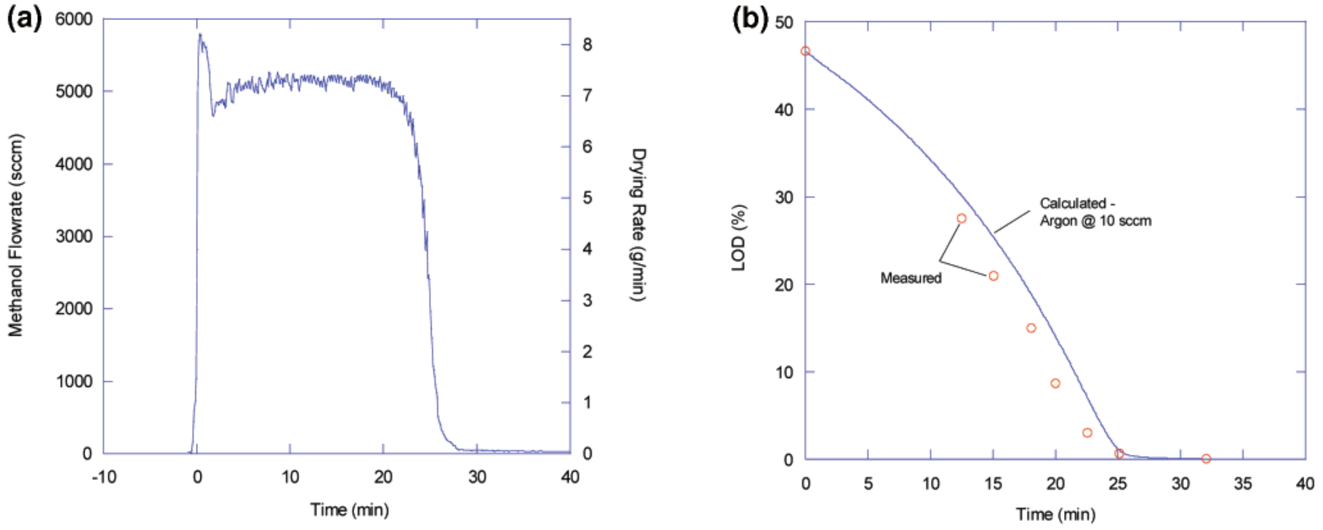

Figure 5. **(a) Microwave vacuum drying profile of methanol (175 g) in $CaCO_3$ (200 g), showing methanol flow rate (left axis) and methanol drying rate (right axis) profiles. The profiles are based on a microwave power input of 150 W, a vacuum pressure of 100 mbar, an oven air temperature of 35 C, and an argon reference gas flow of 10 sccm. Methanol flowrate and drying rate profiles are computed via eqs 6 and 7, respectively. (b) Microwave vacuum drying profile of methanol (175 g) in $CaCO_3$ (200 g), showing calculated and measured LOD%. The profiles are based on an argon reference gas flow of 10 sccm. LOD is computed via eqs 8 and 9.**

shows that while hexane and ethyl acetate are initially removed at a constant drying rate (0−5 min), the hexane profile then decreases at approximately the same rate that ethyl acetate is increasing (5−18 min). These results demonstrate that this method is a valid technique for generating LOD profiles and for determining microwave drying behavior for multiple solvent wet cakes.

3.2. On-Line Flow/Drying Rate Profiling Using Method 1. A gas standard mixture of 1% methanol (mol %) in argon was utilized to obtain a methanol normalization factor (NF_{MeOH}) to determine methanol flowrate and drying rate profiles via on-line MS response. Figure 5a shows the resulting on-line methanol flowrate and drying profiles from the microwave vacuum drying of methanol in calcium carbonate. Using eqs 6 and 7, the methanol flowrate was calculated to be approximately 5000 sccm during the constant rate period, corresponding to a drying rate of approximately 7.5 g/min (Figure 5a). The total amount of recovered methanol was calculated at 179 g, which represents a 2% difference when compared to the initial amount of methanol (175 g) added to the solids.

Figure 5b shows the calculated (eq 9) and measured LOD profiles of methanol in calcium carbonate. The calculated LOD profile is shown to correlate well with the measured LOD, demonstrating that this is a valid technique for generating moisture content profiles for microwave drying. The 1−2 min lag in the calculated LOD profile is due to the previously discussed headspace effects.

3.3. Characteristic Drying Curves for Various Solids. A plot of the drying rate versus the moisture content (LOD) is called the characteristic drying rate curve.[6] Figure 6 illustrates the characteristic drying curves for sand, calcium carbonate, starch, cellulose, and silica gel in methanol calculated using Method 2 with an argon flowrate of 100 sccm. For calcium carbonate and sand, a majority of the drying occurs within the constant rate regime; cellulose and starch display a shorter constant rate drying period; and silica gel has the shortest constant rate period.

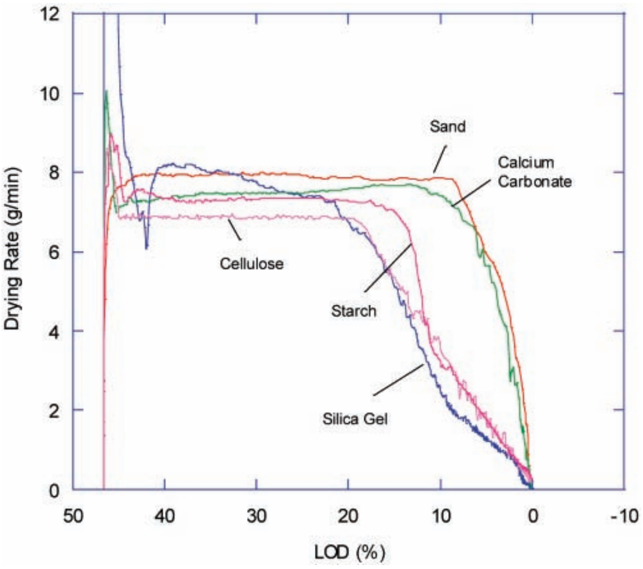

Figure 6. **Characteristic drying rate curves for calcium carbonate, sand, starch, cellulose, and silica gel. The profiles are estimated via Method 2, with an argon flow rate of 100 sccm. Drying starts on the left (LOD = 46.7%) and proceeds to the right.**

The critical moisture content (LOD_c), defined as the transition of the constant rate to the falling rate period, was estimated for each solid from Figure 6. Silica gel, with an LOD_c of 22%, is highest due to its ability to retain moisture within the solid pores (i.e., hygroscopic properties), leading to a lower microwave adsorption efficiency and associated drying rate. Conversely, sand and calcium carbonate have LOD_c's of 8% and 12%, respectively, and do not show the

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(6) *Mujumdar's Practical Guide To Industrial Drying: Principles, Equipment and New Developments*; Devahastin, S., Ed.; Exergex Corporation: Montreal, Canada, 2000; pp 1−20.

Table 1. **Microwave vacuum drying rate of methanol in various materials**

material	average drying rate[a] (g/min)	critical moisture content, LOD_c (%)
sand	7.6	8
calcium carbonate	7.0	12
starch	5.6	15
cellulose	5.5	18
silica gel	4.2	22

[a] Average drying rate = solvent input/drying time. Drying time estimated as duration from initiation of MW power to leveling of recovered liquid, reflected power profiles. Microwave power input = 150 W, oven temperature = 35 °C, vacuum level = 100 mbar.

ability to retain moisture such that the majority of the drying occurs during the constant rate period. Cellulose and starch both resulted in essentially the same average drying rate with LOD_c's of 18% and 15%, respectively; however starch displayed a slightly longer constant rate drying period.

The microwave drying of solids which have a lower critical moisture content will typically result in a higher drying rate,[7] as more drying will take place during the constant rate period. It has also been reported that the dielectric loss factor and corresponding microwave drying rate decreases with increasing hygroscopicity.[8] The average drying rates calculated from the data are shown in Table 1, and support these conclusions. Additional studies with water in various solids resulted in the same order of decreasing microwave drying rates from sand to silica gel, demonstrating that the area method is valuable for estimating the characteristic drying rate curves of solvent wet cakes.

4. Conclusions

Three methods were developed which enabled the calculation of LOD and drying profiles from MS data. Using these methods, LOD profiles were calculated for single and multiple solvent wet cakes, and characteristic drying curves were determined for methanol with several common solids. The microwave vacuum drying rate was found to increase inversely with hygroscopicity in the following order: sand (nonhygroscopic), calcium carbonate, starch, cellulose, and silica gel (hygroscopic). Although Method 1 involves slightly less development, Methods 2 and 3 can be used when calibration gas mixtures (e.g., solvent in reference gas) are not readily available. This is particularly applicable in the case of multiple solvent wet cakes, and we have demonstrated this for a mixture of hexanes−ethyl acetate. To calculate LOD's in real time using Method 1 or 3, it should be noted that the software must be able to compute the respective integral equations and the initial liquid and wet cake mass must be known.

The findings of this work have broadened the applications of mass spectrometry for monitoring drying operations in the pharmaceutical industry, such that the use of an on-line method to monitor the dryer effluent should allow plant operators to track the loss of solvent with time and determine when the product is free of solvent, without additional down time for LOD analysis.

Acknowledgment

The following Pfizer personnel are acknowledged for their assistance, guidance, and support: Raymond Bemish and Paul Richards. I would like to thank the vendors of the microwave dryer and mass spectrometry equipment: Michael Scudese (Pro-C-epT) for assistance using Mi-Mi-Pro microwave dryer; Tony Slapikas and Bill Sands (Ametek) for guidance using the Dycor MS; and Stephen Foster (Pfieffer Vacuum) for assistance with the Omnistar MS.

Received for review August 5, 2004.

OP049853S

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