We analyze the evolution of the vertical composition profile during hydrodynamic-evaporative film thinning as it typically occurs during spin casting mixtures of non-volatile solutes and volatile solvents. We assume that the solvent dominates the hydrodynamic-evaporative film thinning. The internal spatio-temporal evolution of the composition is analyzed with a diffusive-advective approach. The analysis provides transparent physical insights into the influence of the experimental conditions on the evolution of the internal composition. We present power laws that link the process control parameters to the composition evolution, process duration, and final solute coverage. The analysis reveals a characteristic Sherwood Number as fundamental process parameter. It identifies for which stages of the process our analysis is quantitatively relevant and discloses the dominance of either diffusion or evaporation. The analysis is valid for dilute solutions e.g., for the deposition of solute (sub)monolayers. But it is also relevant for the deposition of thicker (polymer) films.

Many studies have analyzed this process experimentally \[45\] and theoretically \[1, 46–161\]. Most of them focus on the radial (in)stability of the film \[2–4, 6, 7, 9–11, 13, 17, 21, 26–28, 30, 31, 33–35, 37–39, 43, 45, 50, 52, 58, 65, 70, 81, 89, 98, 105, 122, 125, 155, 156\] and theoretically \[1–4, 7, 10, 12, 13, 16, 18, 20, 25, 29, 30, 32, 40, 44, 46, 51, 52, 54, 55, 60, 61, 70, 71, 73, 75, 76, 78, 80, 83, 85, 91, 94, 97, 99, 100, 103, 106, 109–117, 119, 121, 124, 126, 136, 139, 145, 147, 151, 153, 154, 157, 158, 161\]. Thus their validity ranges are ill-defined and the insight into the general physics of this interesting hydraulic-evaporative process is limited.

Increasingly spin casting is used for fundamental nucleation and growth studies \[163, 164\] and for the deposition of structured (sub)monolayers (particle arrays, “evaporation-induced self-assembly” \[164, 172\]). This means dilute solutions, which behave rather ideally during most of the spin cast process. This is the motivation and basis of our analysis.

In this report we focus on the evolution of the vertical composition profile during the hydrodynamic-evaporative film thinning. To reveal its general aspects we neglect any complicated, non-linear solution behavior. Thus, for the first time palpable, universal relations between the process parameters and the compositional evolution are presented. The key process parameter, its characteristic Sherwood number \(Sh_tr\), is introduced. It is demonstrated that the analysis is indeed quantitatively valid for low solute concentrations but also yields valuable insights into the behavior of higher concentrated so-
Hydrodynamic film thinning \((E = 0, \text{ dashed})\) dominates \(\approx 30\%\) of the spin cast time; \(\approx 70\%\) is evaporation-dominated \((K = 0, \text{ dashed})\). All this agrees well with experimental results (see inset in Fig. 2 and supplement [176]).

II. Solute concentration evolution — During spin casting, non-volatile solute enriches at the free surface (where the solvent evaporates) and migrates into the film via diffusion. The spatio-temporal evolution of the solute concentration \(c\) is described by

\[ \partial_t c = D \partial_z^2 c + K z^2 (3h - z) \partial_z c, \]  

with boundary conditions

\[ D \partial_z c \big|_{z = h} = E c \big|_{z = h}, \quad \partial_z c \big|_{z = 0} = 0. \]  

The advective term in Eq. 8 is derived from the radial velocity field \(u(r, z)\) of a Newtonian fluid rotating with its solid support (no slip), with a free surface at the top (no stress) [1]:

\[ u(r, z) = 3K r z (h - z/2). \]  

\(r\) and \(z\) are the radial respectively vertical coordinates. The radial volumetric flux \(\phi dz\) is

\[ \phi dz = 2\pi r u(r, z) dz = 6\pi K r^2 z (h - z/2) dz. \]  

With the continuity equation this yields the thinning-induced vertical motion of the horizontal streamlines:

\[ dZ/dt = -1/(2\pi r) \int_z^h \partial_r \phi dz' = -K z^2 (3h - z). \]  

In order to solve Eq. 8 \(h(t)\) is required but not known explicitly, only its inverse, \(t(h)\) (Eq. 5). Since \(h(t)\) respectively \(\tau(\xi)\) are bijective, the time variable can be changed to \(\xi\): \(\partial c = d\tau/dt \cdot d\xi/d\tau \cdot \partial c = -(2E^2 K)^{1/3} (\xi^3 + 1) \partial c\). Rescaling \(y = z/h\) \((y \in [0, 1])\), from substrate to surface) avoids the moving boundary. This leads to:

\[ \partial_\xi c = \frac{\partial_\tau^2 c}{S_{htr} \xi^4 (\xi^3 + 1)} - \left\{ \frac{(\xi y)^2 (3 - y) - y}{2(\xi^3 + 1)} - \frac{y}{\xi} \right\} \partial_y c, \]  

\[ \partial_y c \big|_{y = 1} = S_{htr} \xi c \big|_{y = 1}, \quad \partial_y c \big|_{y = 0} = 0, \]  

where the Sherwood number, \(S_{htr}\), parameterizes the ratio of evaporation to diffusive mass transport on the characteristic length scale of the system, \(h_{tr}\):

\[ S_{htr} = E h_{tr}/D = E^{4/3} (2K)^{-1/3}/D. \]  

By scaling \(c\) with the initial solute concentration \(c_0\) the initial condition is \(c \big|_{\xi \to \infty} = 1\). Thus the system is parametrized completely by \(S_{htr}\). Eq. 13 can be solved numerically with \(\xi\) as independent variable. Eq. 13 then provides \(\tau\) as function of \(\xi\) i.e., finally, \(c(t, z)\).
III. General aspects of the concentration evolution —
Based on the solution of Eq. (13) the impact of $S_{ht}$ (i.e., of the individual system parameters $K$, $E$, and $D$) is now analyzed. Fig. 3 shows profiles of $c$ during film thinning for $S_{ht}$ larger and smaller than 1 (i.e., convection dominating over diffusion and vice versa) and for film heights larger and smaller than $h_{tr}$, respectively. It reveals the competition between evaporative enrichment, spin-off, and diffusive dilution. In general, larger $S_{ht}$ means more pronounced gradients in $c$. If $h \gg h_{tr}$, solute enrichment occurs only locally near the free surface and $c \approx c_0$ near the substrate. If $h \ll h_{tr}$, $c$ also increases near the substrate. $S_{ht} = 1$ and $h = h_{tr}$ mark the transition: The solute gradient just reaches the film/substrate interface and $c$ just begins to increase globally.

Fig. 4 shows as function of $h/h_{tr}$: A) The total solute amount $N$ per unit area $A$ ($N/A = \int_0^h c\,dz$); B) $c/c_0$ at the surface respectively substrate/film interface; C) the difference between the surface and the substrate/film interface concentrations, $\Delta c/c_0$ i.e., the relative enrichment. Both, $h = h_{tr}$ and $S_{ht} = 1$ mark transitions between distinctly different behaviors. I) For $h \gg h_{tr}$ spin-off dominates film thinning. Therefore, globally $c/c_0$ remains approximately constant and $N/A$ decreases. Nevertheless, there is a surficial solute enrichment due to evaporation. But this $\Delta c/c_0$ only becomes substantial for $S_{ht} > 1$. It has a maximum at $h = h_{peak}$ ($h_{peak} \approx h_{tr}$ for $S_{ht} \approx 1$). II) For $h \ll h_{tr}$, evaporation dominates. Solvent loss leads to increasing $c/c_0$ while $N/A$ and $\Delta c/c_0$ remain constant (because spin-off becomes negligible). Remarkably, for $h < h_{tr}$, $N/(Ac_0h_{tr})$ remains approximately constant and independent from $S_{ht}$ whereas $\Delta c/c_0$ increases with increasing $S_{ht}$.

Fig. 5 shows $c(z = h = h_{tr})/c_0$, the concentration at the free surface for $h = h_{tr}$, and $N(h \to 0)/(Ac_0h_{tr})$, the rescaled final coverage. Both are plotted as function of $S_{ht}$. For $S_{ht} \ll 1$ diffusion dominates and $c$ is mostly homogeneous (Fig. 3). In this case $c_0Et_{sc}$ is the total amount of solute that is not spun off with the solvent. Distributing this into a film with $h_{tr}$ yields $c/c_0 = Et_{sc}/h_{tr} = 2\sqrt{3}/3^{3/2} \approx 1.2$ (Fig. 5). The final solute coverage for $S_{ht} < 1$ is (in agreement with $[8]$)

$$\Gamma = N(h \to 0)/A \approx c_0 h_{tr} \approx 0.8 c_0 (K/E)^{-1/2}.$$  \hspace{1cm} (16)

For $S_{ht} \gg 1$ it is only $\approx 6\%$ lower.

IV. Relative surficial enrichment maximum — Fig. 6 shows $\Delta c_{peak}/c_0$ and its spatio-temporal properties as function of $S_{ht}$. Symbols denote the results from the numerical analysis. The solid lines show power laws for $S_{ht} < 1$ which are rationalized by analyzing the underlying processes.

Panel A) shows $h_{peak}$ rescaled by $h_{tr}$, i.e., the film thickness at which $\Delta c = \Delta c_{peak}$ as function of $S_{ht}$. $\Delta c_{peak}$ emerges from the competition between evaporative enrichment, spin-off, and diffusional equilibration. Evaporation and spin-off dominate at opposite ranges of $h$.

For large $h$, surficial spin-off efficiently suppresses enrichment: $\Delta c_{peak}$ requires $2K h_{peak}^2 < E$ (from Eq. 1). However, for small $h$, diffusional equilibration is fast, so
\[
\Delta c_{\text{peak}} \text{ requires } D/h_{\text{peak}} < E. \text{ Optimizing both conditions simultaneously (} E \text{ is linear and protagonistic in both cases, and therefore cancels out) reveals the same power law as numerics (which supplies the prefactor):}
\]
\[
h_{\text{peak}} \approx (D/K)^{1/4} \approx 1.2 h_{tr} S_{tr}^{-1/4}. \quad (17)
\]

Panel B) shows the time \( t_{\text{peak}} \) rescaled by \( t_{\text{sc}} \) i.e., the time at which \( \Delta c = \Delta c_{\text{peak}} \). Before reaching \( h_{\text{peak}} \), thinning is dominated by spin-off. Hence \( t_{\text{peak}} \) can be estimated by inserting \( h_{\text{peak}} \) into \( h = (2Kt)^{-1/2} \) (the solution to Eq. 1 with \( E = 0 \)):
\[
t_{\text{peak}}/t_{\text{sc}} \approx 0.31 E^{2/3} K^{-1/6} D^{-1/2} \approx 0.35 \sqrt{S_{tr}}. \quad (18)
\]

Panel C presents \( \Delta c_{\text{peak}}/c_0 \), reflecting the balance between evaporative enrichment and diffusive equilibration i.e., \( S_{tr}^{h_{\text{peak}}} = E h_{\text{peak}}/D = S_{tr}^{3/4} \). With Eq. 17 this means:
\[
\Delta c_{\text{peak}}/c_0 \approx 0.46 E K^{-1/4} D^{-3/4} \approx 0.55 S_{tr}^{3/4}. \quad (19)
\]

**Discussion and Conclusions** — Section I introduces the system-specific fundamental length and time scales \( (h_{tr}, t_{\text{sc}}) \) for the spin casting process of an ideal Newtonian volatile liquid. These reduce the general spin cast equation (Eq. 1) to its fundamental form [146] and lead to a universal film thinning behavior (Eq. 5). The total spin coating time, \( t_{\text{sc}} \) is calculated (Eq. 3) as function of the process parameters \( (E, K, D) \). For any combination of these parameters, in the first 30% of the process time, thinning is governed by hydrodynamics. During 70% of the time evaporative thinning dominates until complete drying.

Sections II through IV analyze the spin cast process of a mixture of a volatile solvent and a non-volatile solute assuming constant process parameters \( E, K, \) and \( D \).

It is found that the spatio-temporal evolution of the solute concentration within the thinning film is universally characterized by a Sherwood number, \( S_{tr} \), scaled to the system-inherent fundamental length, \( h_{tr} \) (it reflects the competition between evaporative solute enrichment and diffusional dilution at \( h_{tr} \)). For \( S_{tr} < 1 \) (diffusion dominates) the spatio-temporal occurrence of the relative surficial enrichment maximum is related to the process parameters via universal power laws (Eqs. 17 [18] and [19] see Fig. 6). These findings are rationalized semi-quantitatively with the underlying physics. At last, the final solute coverage \( \Gamma \) is calculated from the process parameters (Eq. 16).

To examine the relevance of our analysis for real cases, where \( E, K, \) and \( D \) are not necessarily always constant (e.g. depending on the solute concentration), we assume a typical solvent (e.g. toluene) with molar mass \( M_s \approx 0.1 \text{ kg/mol} \), density \( \rho_0 \approx 10^3 \text{ kg/m}^3 \), \( \nu = 6 \cdot 10^{-7} \text{ m}^2\text{s}^{-1} \), and \( E = 10^{-6} \text{ m/s}^{1/2} \) [18]. We consider two examples: A) A typical polymer solution [162]; B) A nanoparticle solution for submonolayer deposition.

Case A): Polymer with \( M_p = 100 \text{ kg/mol} \), \( 
\rho_v = 10^3 \text{ kgm}^{-3} \), and \( c_{0,p} = 10 \text{ kgm}^{-3} \) (i.e., mole fractions \( x_{0,p} = 10^{-6} \) and \( x_{0,b} \approx 1 \)), \( D_p = 10^{-11} \text{ m}^2\text{s}^{-1} \) [172] and \( K = 10^{11} \text{ m}^{-2}\text{s}^{-1} \) (\( \approx 4000 \text{ rpm} \)). This yields: \( h_{tr} \approx 1.7 \cdot 10^{-6} \text{ m} \) (Eq. 3), \( S_{tr} \approx 0.17 \) (Eq. 15), \( c_p(h_{tr})/c_{0,r} \approx 1.2 \) (Figs. 3 and 6) and \( \Gamma_p \approx 17 \cdot 10^{-6} \text{ kgm}^{-2} \) (Eq. 19) i.e., a final film thickness of \( \approx 17 \text{ nm} \). This is in reasonable agreement with experimental results [162].

Case B): Spheres with radius \( r_{\text{NP}} = 36 \text{ nm} \). \( D_{\text{NP}} = 10^{-11} \text{ m}^2\text{s}^{-1} \) (estimation: Stokes-Einstein) as in case A) and thus \( h_{tr} \) and \( S_{tr} \) are identical to case A). A 50% monolayer coverage means \( \Gamma_{\text{NP}} \approx 10^{14} \text{ mol}^{-2} \) i.e., \( c_{0,NP} \approx 5 \cdot 10^{-5} \text{ mol/m}^3 \) \( x_{0,NP} \approx 5 \cdot 10^{-9} \) \( x_{0,b} \approx 1 \) with Eq. 16.

In both cases the initial solute mole fraction is small. Therefore film thinning to \( h_{tr} \) will follow Eq. 1 with \( K \) and \( E \) remaining approximately constant because thinning is hydrodynamically dominated and the solute concentration barely changes. Hence \( \Gamma \) can be calculated with Eq. 16 which is not affected by changes in \( K \) and \( E \) occurring after reaching \( h_{tr} \).

For both examples \( S_{tr} < 1 \). Therefore our analysis regarding \( h_{\text{peak}}, \ t_{\text{peak}}, \ \text{and } \Delta c_{\text{peak}} \) is relevant even quantitatively because all three maxima occur at \( h > h_{\text{peak}} \) i.e., the (surficial) solute concentration has not yet increased substantially above \( c_0 \) (Figs. 5 and 6). Accordingly, also Eqs. 17 [18] and [19] are applicable.

Of course, evaporative film thinning at \( h < h_{\text{peak}} \) will eventually increase the solute concentration and thus change \( E \) and \( K \). However, in particular for "evaporation-structured" submonolayer particle array deposition, non-ideality will only become relevant for \( h \ll h_{tr} \), when \( h \) is already in the range of \( r_{\text{NP}} \), because
typically $x_{0,\text{NP}} \ll 1$. For low solubilities our analysis reveals quantitatively whether solute aggregation occurs first at the top surface (if evaporative solute enrichment dominates) or globally homogeneous (or heterogeneously at the substrate) if diffusive equilibration is more efficient at the corresponding film thickness. Last not least, our analysis predicts a quantifiable behavior based on measurable parameters of the initial solution. In particular the predicted $\Gamma$ and $h(t)$ \cite{18} are easily measurable. Thus not only the validity of the approach can be evaluated. It is possible to specifically address the influence of individual parameters of the multi-parameter spin casting process. For instance, parameters not related to mixing can be probed, because a sufficient reduction of $c_0$ will per definition render the solution behaving approximately “ideally”. All this proposes our approach as a future basis for incorporating specific (non)-linear properties of specific systems. Due to the predominantly analytic approach such a refined approach still will reveal general physical insights.

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\* Electronic address: stefan.karpitschka@mpikg.mpg.de

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[174] except for [86, 146], which reveal very little about the compositional evolution.
[175] The expression Eq. (43) presented in [79] is not correct!
[176] See supplemental material at http://link.aps.org/supplemental/... for experimental thinning curves, a step-by-step derivation of Eq. 13 and details on the numerical methods.