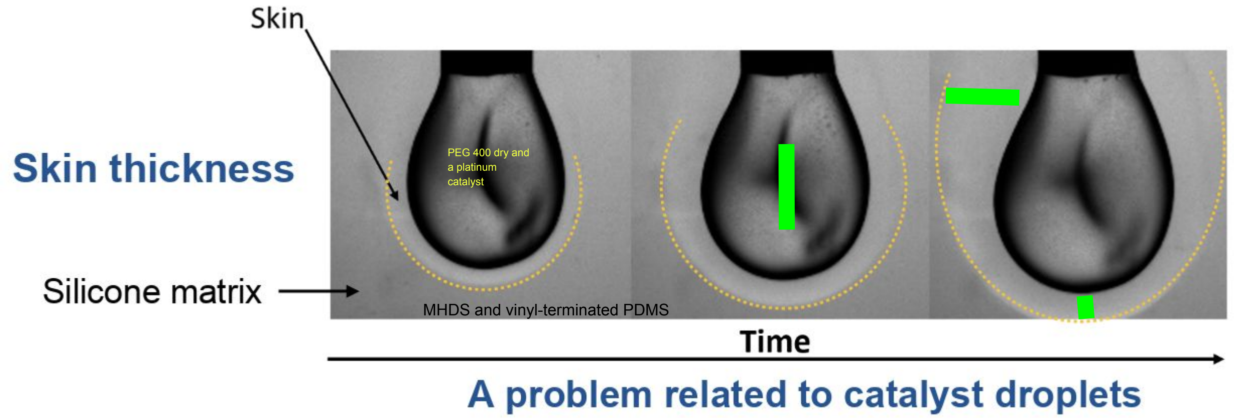
Faculté
de physique et ingénierie
Université de Strasbourg

Characterization of Wrinkle Formation in PEG-Silicone Emulsions

Adeildo Santos de Jesus
Institution: University of Strasbourg
Supervisor: Leandro Jacomine and Wiebke Drenckhan
Charles Sadron Institute - MIM - Strasbourg

Previous works

The research group Mousse at ICS was investigating the basic physics and chemistry of a drop containing PEG and a platinum catalyst that in contact with silicone environment it forms a capsule. During the study, they observed the wrinkling and skin formation of this capsule.



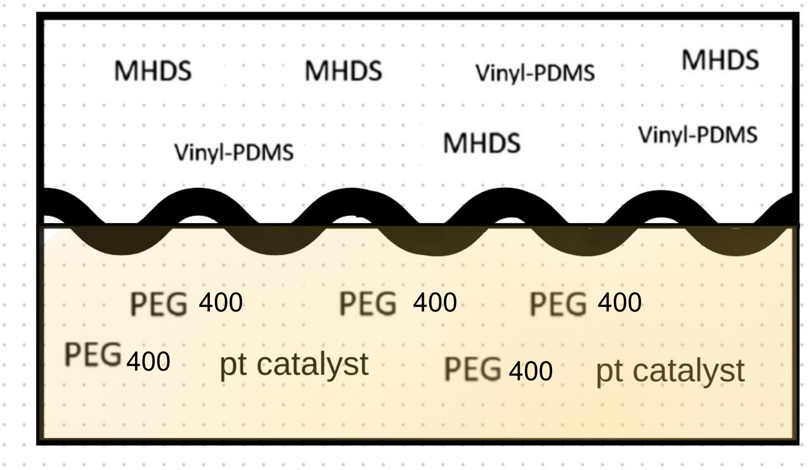
wrinkles and deformations in the silicone skin indicate internal forces

Potential Applications:

- Drug delivery in the body
- use in perfumes
- material for creating cartilage

Setup for observing wrinkle formation in PDMS layers catalyzed by Pt

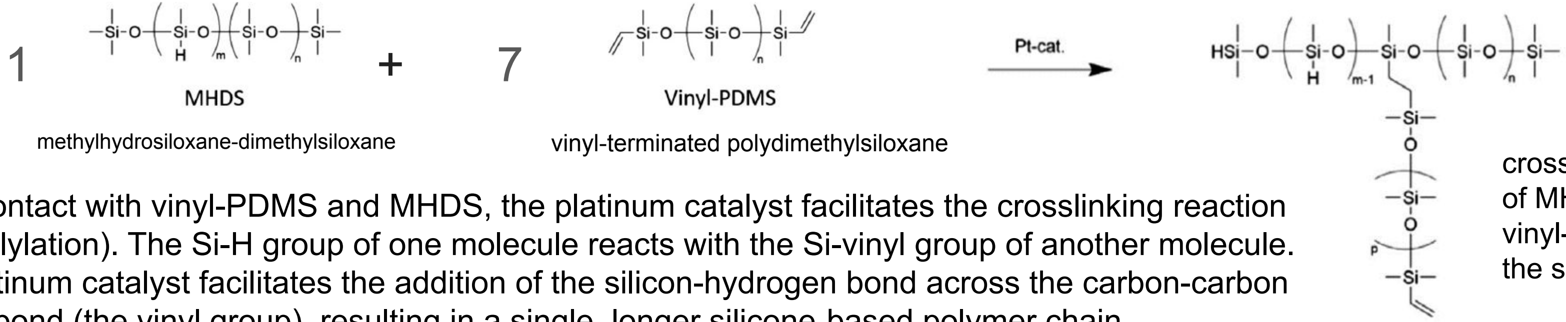
Diagram of the biphasic Petri dish setup



Components:

- **Bottom half:** PEG 400 dry and a platinum catalyst
- **Top half:** MHDS and vinyl-terminated PDMS

The platinum catalyst rises due to its lower density. The displacement of the catalyst is controlled by Brownian motion and gravitational force.

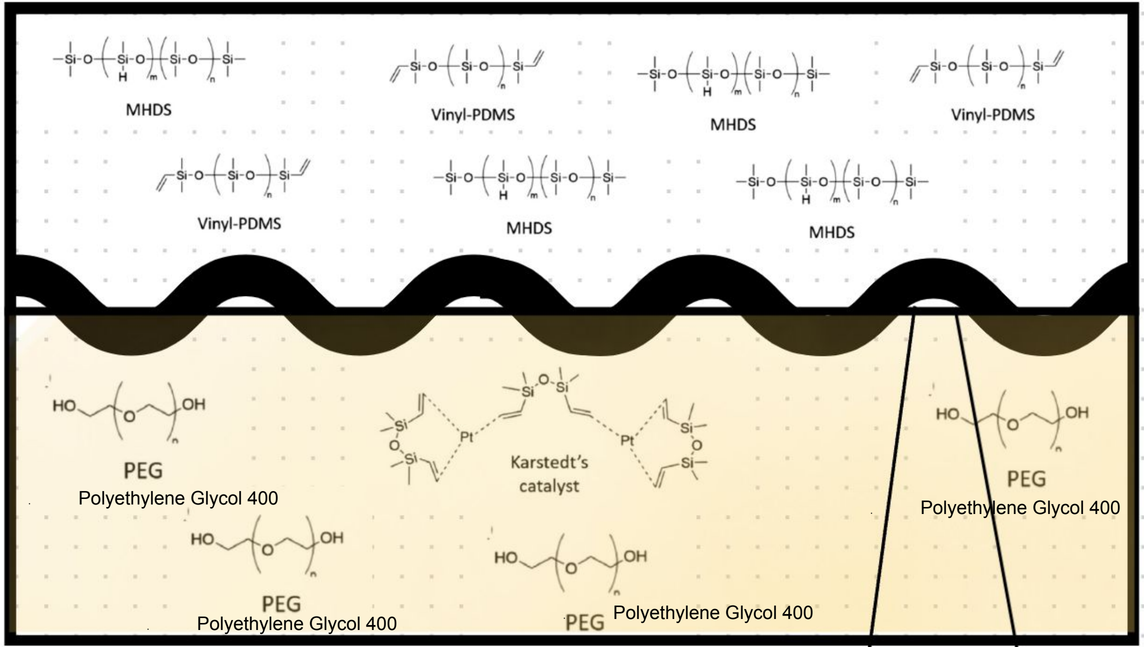


methylhydrosiloxane-dimethylsiloxane

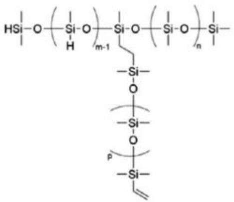
vinyl-terminated polydimethylsiloxane

crosslinking reaction of MHDS with vinyl-PDMS to form the skin

Upon contact with vinyl-PDMS and MHDS, the platinum catalyst facilitates the crosslinking reaction (hydrosilylation). The Si-H group of one molecule reacts with the Si-vinyl group of another molecule. The platinum catalyst facilitates the addition of the silicon-hydrogen bond across the carbon-carbon double bond (the vinyl group), resulting in a single, longer silicone-based polymer chain.

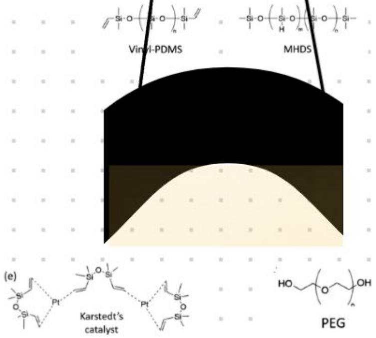


longer silicone-based polymer chain



Crosslinking reaction

As the skin forms, this surface begins to exhibit certain wrinkles

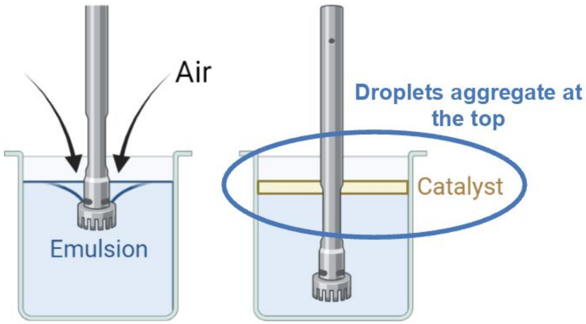


Internship Objectives

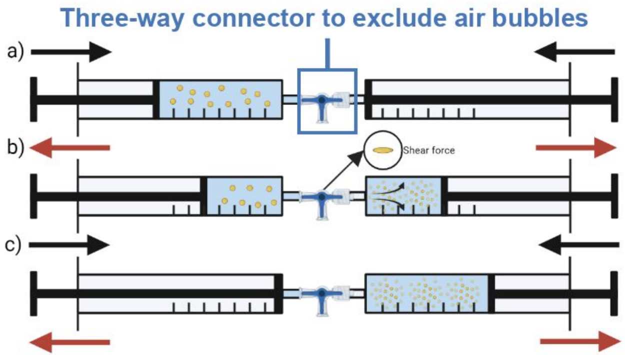
To quantify the additional material resulting from wrinkle formation in PEG-silicone emulsions within Petri dishes and to determine the internal stresses during the skin growth process.

Experimental procedures

To avoid this problem



Introduction of air bubbles via vortex generated by Ultra-Turrax

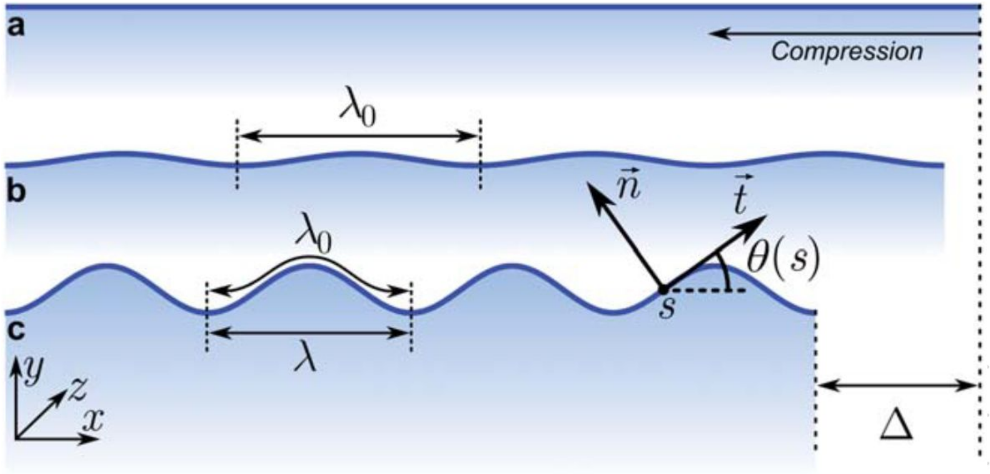


Premixing Double-Syringe setup

To prevent side reactions, PEG-400 is dried under vacuum at 40°C and stored under argon to avoid air exposure.

The silicone mixture with a Pt-catalyst (0.3%) is added to the PEG. A double-syringe system pre-mixes the phases to reduce bubbles, followed by an Ultra-Turrax mixer to ensure a homogeneous emulsion, minimizing air inclusion.

Modeling Wrinkle Formation based on the work of Brau (2013)



When a surface is compressed, it experiences a force that it resists due to its stiffness, described by its **bending modulus B*.**

This force leads to a state where the surface seeks to minimize its potential energy, leading to the formation of wrinkles with a **characteristic wavelength, λ_0.**

The sequence from (a) to (c) shows the progressive wrinkle development under compression, from initial flat state (a) to fully developed wrinkle pattern (b)

The wavelength can be predicted using the formula : $$\lambda_0 = 2\pi\left(\frac{3B}{E_s}\right)^{1/3},$$

Where $\mathbf{E}_s$ is the Young's modulus of the substrate.

*F. Brau et al. Wrinkle to fold transition: influence of the substrate. Soft Matter, 9(24):8177–8186, 2013.

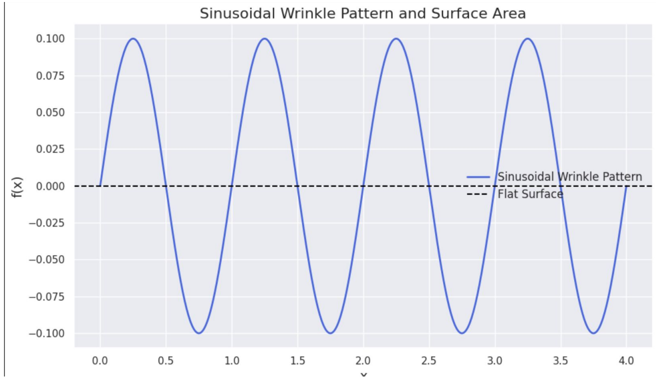
Mathematical model based on the creation of undulations on a surface

Sinusoidal Wrinkle Model: $f(x) = A \sin\left(\frac{2\pi}{\lambda} x\right)$

Here, A represents amplitude, and λ wavelength.

We integrate along the curve: $\text{Curve Length} = \int_0^{\lambda} \sqrt{1 + \left(\frac{df}{dx}\right)^2}\, dx$, where $\frac{df}{dx} = \frac{2\pi A}{\lambda} \cos\left(\frac{2\pi}{\lambda} x\right)$

So, the $\text{Curve Length} = \int_0^{\lambda} \sqrt{1 + \left(\frac{2\pi A}{\lambda} \cos\left(\frac{2\pi}{\lambda} x\right)\right)^2}\, dx$



Exploratory Phase: Initial Wrinkle Formation Observations





Initial Observations:

A smooth layer formation, which gradually accumulated more material, leading to wrinkle formation.

As the skin formed and grew under compression, the observed wrinkles' pattern and frequency changed.

Key Observations:

- Square Geometry:
  Two preferential directions of compression along the edges.
  A singularity at 45 degrees along the diagonals.

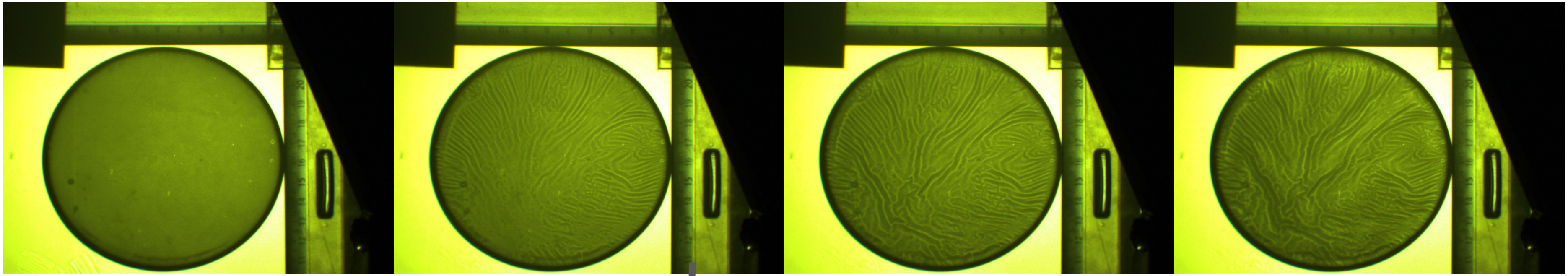
- Circular Geometry:
- Compression occurs equally in all directions.
- Reduced edge effects; in the middle, stresses come from everywhere.

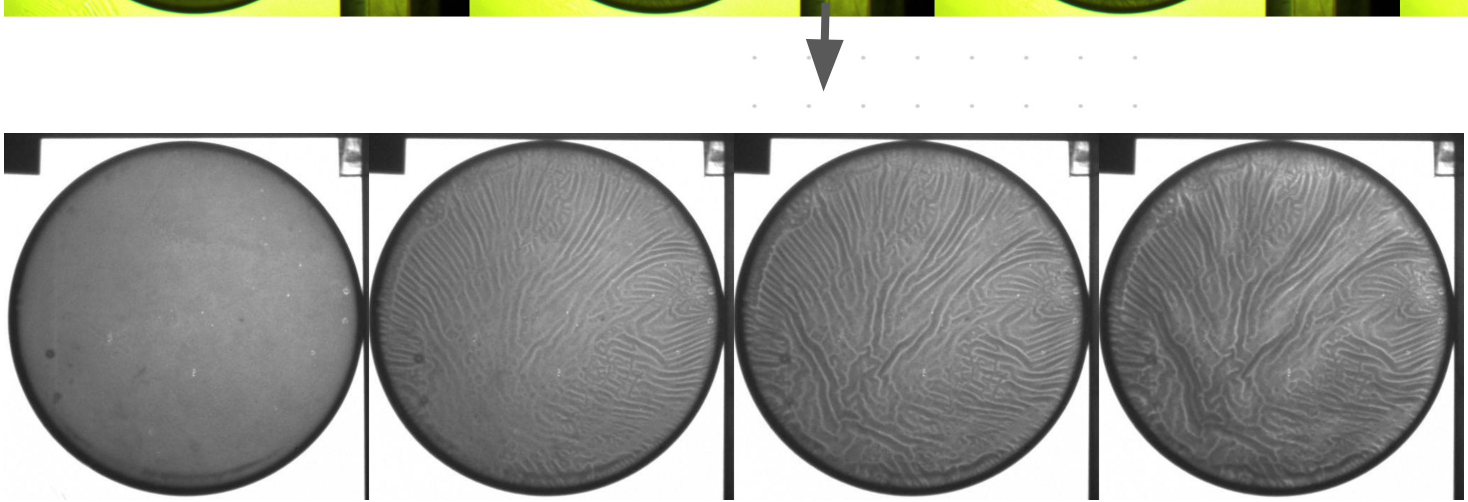
Results:

- Round geometry reduced edge effects, ensuring uniform stress distribution.

Pre-Treatment Phase: Visual Progression of Wrinkle Patterns

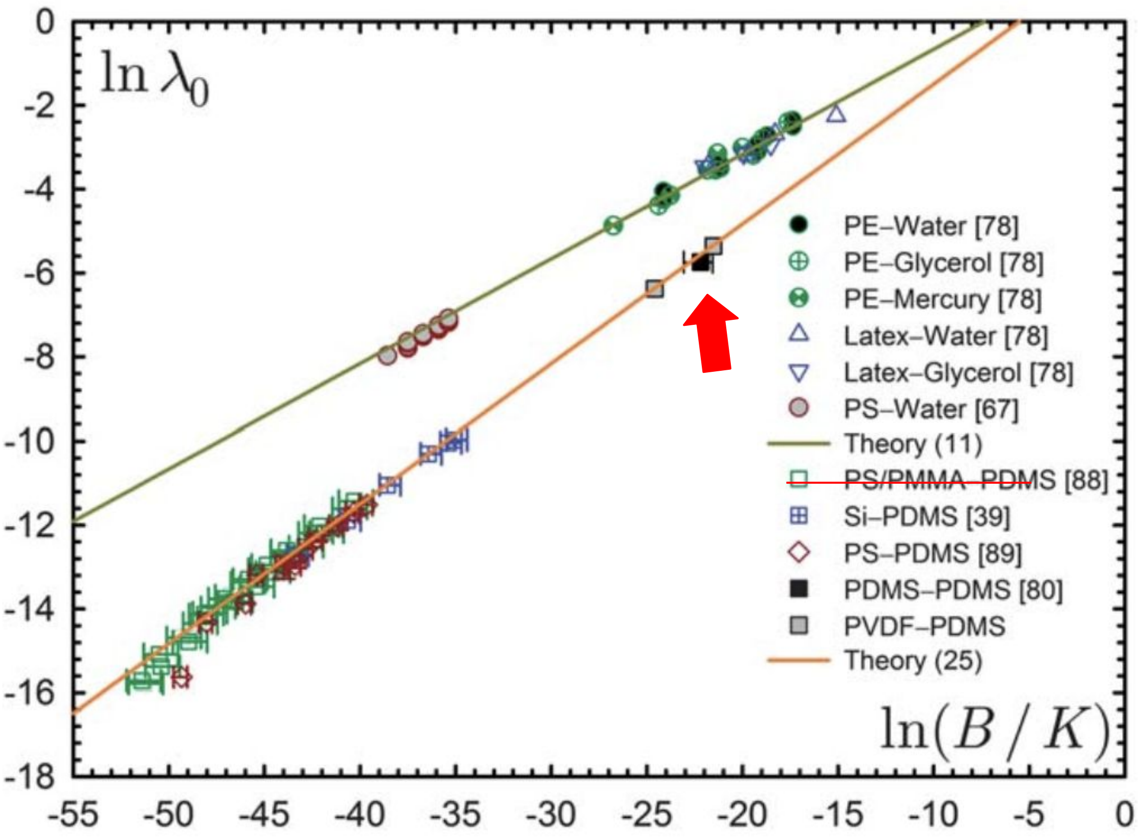
The stages of wrinkle evolution in our PEG-Silicone emulsions





The initial moments are crucial in wrinkle formation, setting the stage for the final patterns. Internal contractions quickly shape these early structures, guided by the material's urge to reduce energy under stress.

The graph shows ln(λ_0) versus ln(B/K) for different materials.



To obtain the bending modulus **B**, we analyzed data from the article 'Wrinkle to Fold Transition: Influence of the Substrate' published in Soft Matter, 2013.In this graph, the author obtained different values of B for different materials.

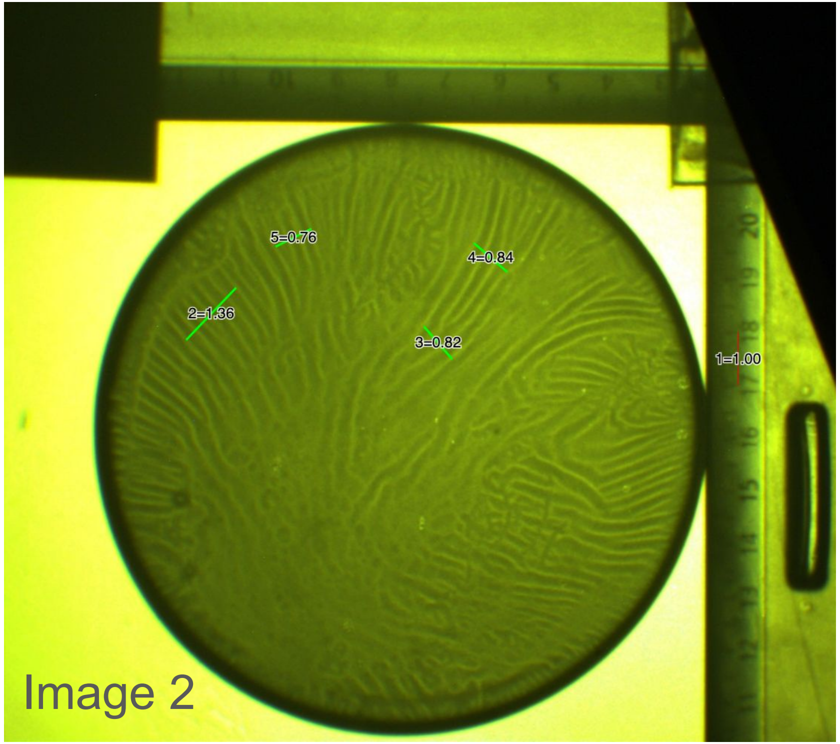
$$\lambda_0 = 2\pi \left(\frac{3B}{E_s}\right)^{1/3} = 0.38 \pm 0.14\,\text{cm}$$

$$K = E_s/3$$ (Effective Stiffness)

*Brau et al. Wrinkle to fold transition: influence of the substrate. Soft Matter, 9(24):8177–8186, 2013

Utilizing the PhotoMeasure tool for distance measurements between wrinkles

Detailed view of wrinkle formations with annotated distances



Wrinkle ID	Number of Wrinkles	Average Distance (cm)	Uncertainty (cm)
ID 2	6	0.2267	± 0.028
ID 3	3	0.2733	± 0.028
ID 4	3	0.2800	± 0.028
ID 5	3	0.2533	± 0.02
Total	15	-	-

Average Distance per Wrinkle: (0.2408, ± 0,014) cm

$$\text{Mean Distance} = \frac{\sum \text{Distances}}{\text{Number of Distances}}$$

Standard Deviation:

$$\text{SD} = \sqrt{\frac{\sum (X_i - \text{Mean})^2}{N-1}}$$

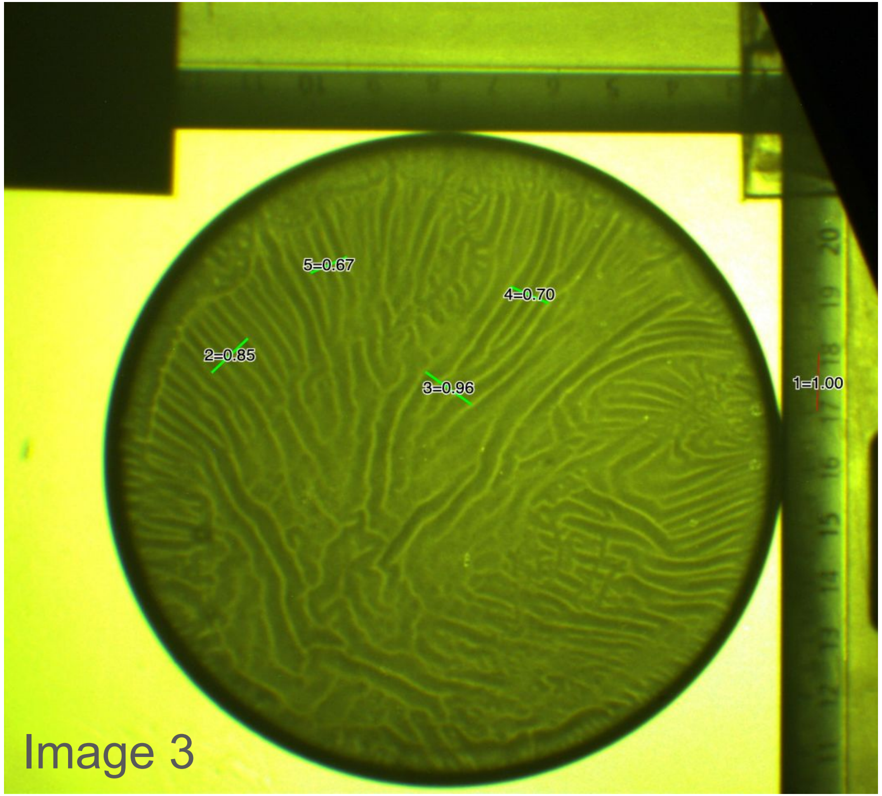
Standard Error of the Mean:

$$\text{SEM} = \frac{\text{SD}}{\sqrt{N}}$$

Tool:
https://eleif.net/photomeasure#

Utilizing the PhotoMeasure tool for distance measurements between wrinkles

Detailed view of wrinkle formations with annotated distances



Wrinkle ID	Number of Wrinkles	Average Distance (cm)	Uncertainty (cm)
ID 2	4	0.2125	± 0.024
ID 3	3	0.320	± 0.024
ID 4	3	0.233	± 0.024
ID 5	3	0.223	± 0.024
Total	13	-	-

Average Distance per Wrinkle: (0.2471 ± 0,025) cm

$$\text{Mean Distance} = \frac{\sum \text{Distances}}{\text{Number of Distances}}$$

Standard Deviation:

$$\text{SD} = \sqrt{\frac{\sum (X_i - \text{Mean})^2}{N-1}}$$

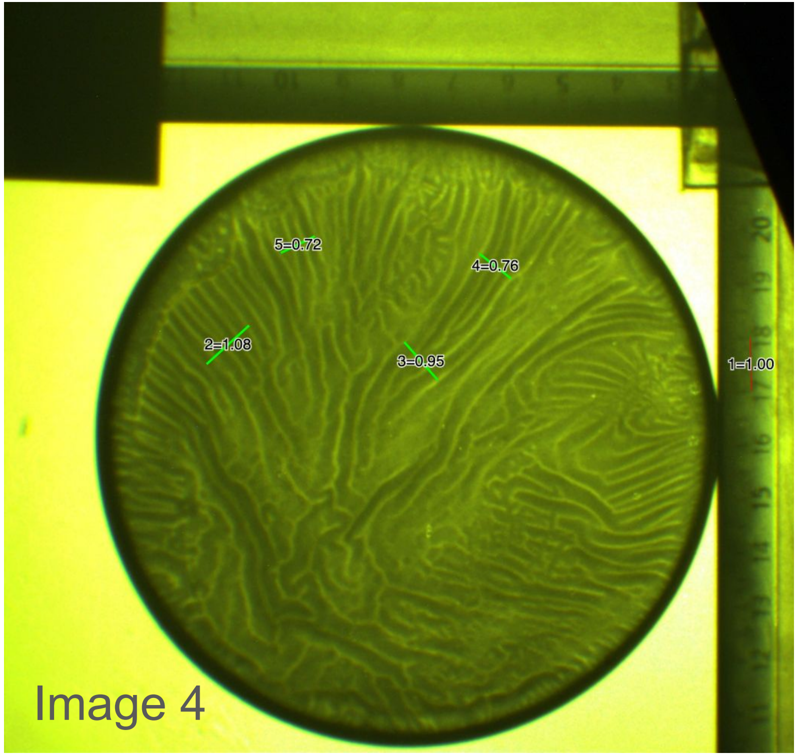
Standard Error of the Mean:

$$\text{SEM} = \frac{\text{SD}}{\sqrt{N}}$$

Tool:
https://eleif.net/photomeasure#

Utilizing the PhotoMeasure tool for distance measurements between wrinkles

Detailed view of wrinkle formations with annotated distances



Wrinkle ID	Number of Wrinkles	Average Distance (cm)	Uncertainty (cm)
ID 2	3	0.270	0.015
ID 3	3	0.320	0.015
ID 4	3	0.253	0.015
ID 5	3	0.240	0.015
Total	12	-	-

Average Distance per Wrinkle: (0.271 ± 0.015) cm

$$\text{Mean Distance} = \frac{\sum \text{Distances}}{\text{Number of Distances}}$$

Standard Deviation:

$$\text{SD} = \sqrt{\frac{\sum (X_i - \text{Mean})^2}{N - 1}}$$

Standard Error of the Mean:

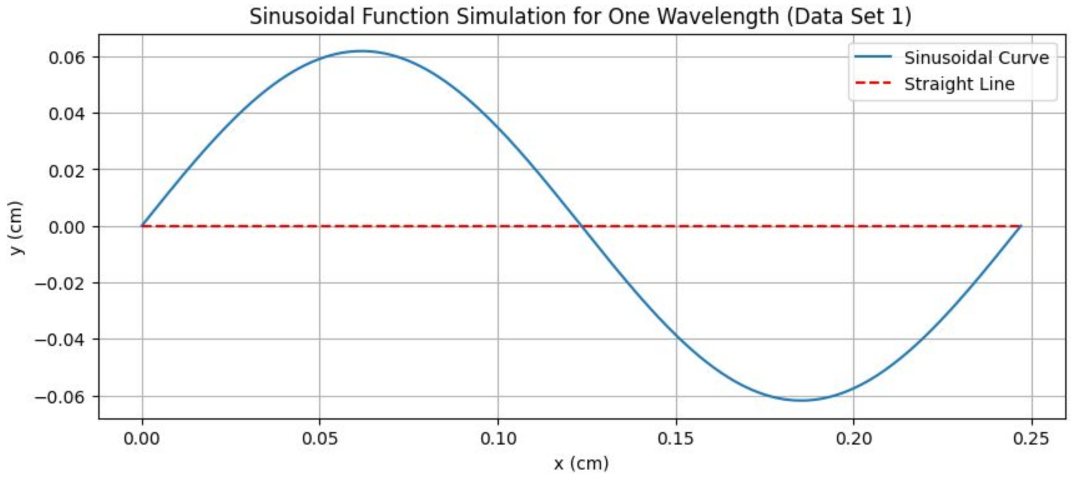
$$\text{SEM} = \frac{\text{SD}}{\sqrt{N}}$$

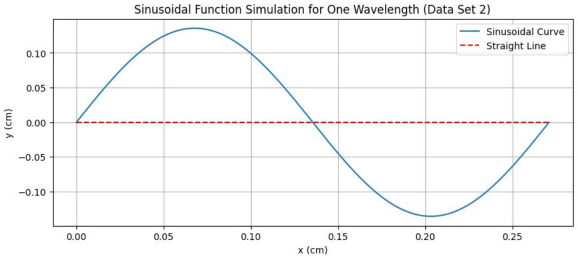
Tool:
https://eleif.net/photomeasure#

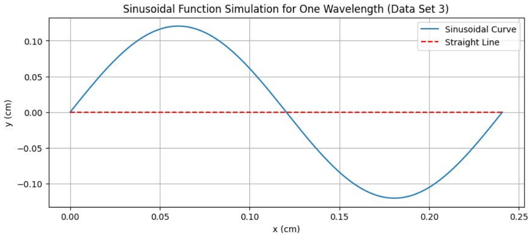
Data Set: Data Set 1
Arc Length of the Sinusoidal Curve: 0.3621 cm
Straight Line Length: 0.2471 cm
Extra Length Due to the Curve: 0.1150 cm

Data Set: Data Set 2
Arc Length of the Sinusoidal Curve: 0.3972 cm
Straight Line Length: 0.2710 cm
Extra Length Due to the Curve: 0.1262 cm

Data Set: Data Set 3
Arc Length of the Sinusoidal Curve: 0.3529 cm
Straight Line Length: 0.2408 cm
Extra Length Due to the Curve: 0.1121 cm







Comparison of Arc Lengths of Sinusoidal Curves with lambda 0

Arc Length Comparisons:

Data Set	Arc Length of Sinusoidal Curve (cm)
Data Set 1	0.3621
Data Set 2	0.3972
Data Set 3	0.3529

Table: Comparison of Arc Lengths of Sinusoidal Curves

The measured arc lengths fall within the predicted range of lambda 0, supporting the theoretical model's validity.

Equation for λ_0:

$$\lambda_0 = 2\pi \left(\frac{3B}{E_s}\right)^{1/3} = 0.38 \pm 0.14 \text{ cm}$$

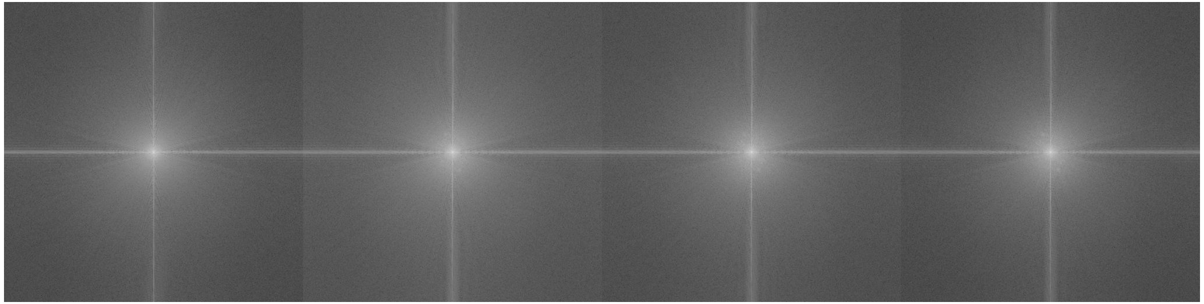
Conclusion

- We could quantified the extra material due to wrinkle formation in PEG-silicone emulsions.
- We determined the internal stresses during the skin growth process.
- We measured Arc Lengths of Sinusoidal Curves:
  Data Set 1: 0.3621 cm
  Data Set 2: 0.3972 cm
  Data Set 3: 0.3529 cm
- Theoretical Prediction:
  Lambda = 0.38 ± 0.14 cm
  The measured values fall within the theoretical range.
- This alignment supports the validity of the theoretical model used to describe wrinkle formation.

Future possibilities

Fourier Transform Analysis:
- Use Fast Fourier Transform (FFT) to analyze wrinkle patterns.
- Identify dominant spatial periods in the wrinkle formation.
- Gain a comprehensive understanding of wrinkle distribution.

3D Analysis:
- Extend analysis to 3D models.
- Apply FFT in 3D to determine spatial periods and stress distributions.
- Compare 3D results with 2D analyses to identify additional factors.



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Thank you

Bending Energy of the Film

The bending energy per unit length U_B is given by:

$$U_B = \frac{B}{2} \int \left(\frac{d^2 y}{dx^2} \right)^2 dx$$

where B is the bending modulus and $y(x)$ describes the deflection of the film.

Deformation Energy of the Substrate

The deformation energy per unit length U_S is proportional to the Young's modulus of the substrate E_s and the displacement $y(x)$:

$$U_S = \frac{E_s}{k^2}$$

Total Energy

The total energy is the sum of the bending energy of the film and the deformation energy of the substrate:

$$U_{\text{total}} = U_B + U_S$$

Substituting the expressions for U_B and U_S:

$$U_{\text{total}} = \frac{Bk^2}{2} + \frac{E_s}{k^2}$$

Minimizing the Total Energy

To find the characteristic wavelength λ_0, we minimize the total energy with respect to the wavenumber k:

$$\frac{dU_{\text{total}}}{dk} = Bk - \frac{2E_s}{k^3} = 0$$

Relationship Between k and λ_0

The relationship between the wavenumber k and the characteristic wavelength λ_0 is:

$$\lambda_0 = \frac{2\pi}{k}$$

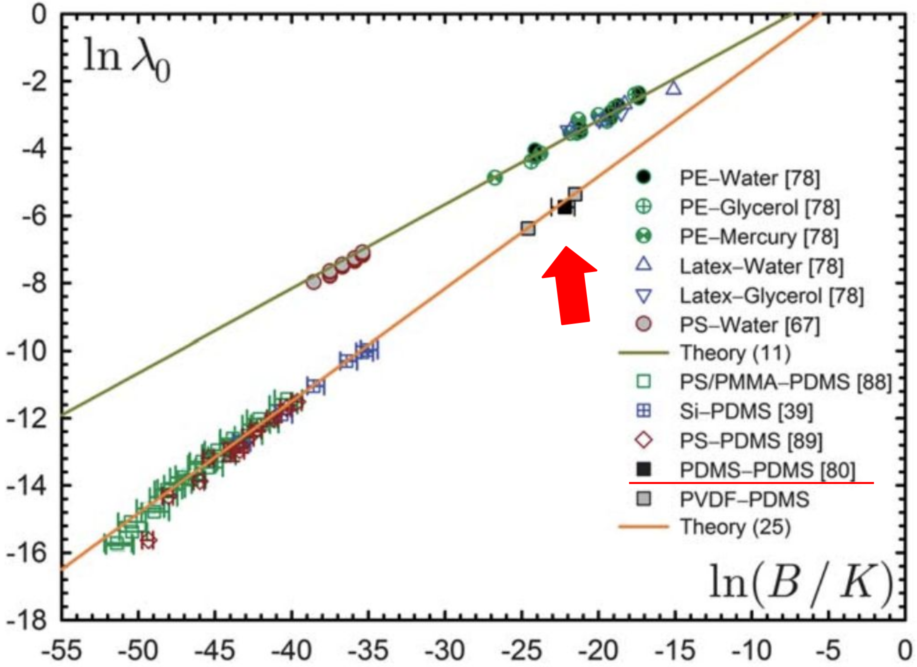
Substituting the expression for k:

$$\lambda_0 = \frac{2\pi}{\left(\frac{2E_s}{B}\right)^{1/4}}$$

Simplifying, we get:

$$\lambda_0 = 2\pi \left(\frac{B}{2E_s}\right)^{1/4}$$

The graph shows ln(λ_0) versus ln(B/K) for different materials.



To obtain the bending modulus **B**, we analyzed data from the article 'Wrinkle to Fold Transition: Influence of the Substrate' published in Soft Matter, 2013.In this graph, the author obtained different values of B for different materials.

$K = E_s/3$ (Effective Stiffness)

$$\lambda_0 = 2\pi\left(\frac{3B}{E_s}\right)^{1/3} = 0.38 \pm 0.14\,\text{cm}$$

To measure the bending modulus B for the crosslinking reaction, the material was treated with ultraviolet radiation in the presence of oxygen, generating ozone. This treatment increased the crosslink density and rigidity of the PDMS surface, forming a brittle layer covalently bonded to the underlying uncured elastomer. The treated samples were then subjected to controlled compression using a custom-built stretching-compressing device, allowing precise measurements of deformation under applied stress.

*Brau et al. Wrinkle to fold transition: influence of the substrate. Soft Matter, 9(24):8177–8186, 2013

Given values:

$$E = 25 \times 10^3\,\text{Pa} \quad \text{(Young's modulus)}$$

$$K = \frac{E}{3} = \frac{25 \times 10^3}{3} = 8333.33\,\text{Pa}$$ (Effective Stiffness)

$$\ln\left(\frac{B}{K}\right) = -22.1$$

Calculate $\frac{B}{K}$:

$$\ln\left(\frac{B}{K}\right) = -22.1$$

$$\frac{B}{K} = e^{-22.1}$$

Evaluate the exponential:

$$e^{-22.1} \approx 2.669 \times 10^{-10}$$

Solve for B:

$$B = K \times e^{-22.1}$$

$$B = 8333.33 \times 2.669 \times 10^{-10}$$

$$B \approx 2.224 \times 10^{-6}\,\text{Pa}$$

Brau, F., et al. (2010) Multiple-length-scale elastic instability mimics parametric resonance of nonlinear oscillators. Nature Physics, 7, 56-60.

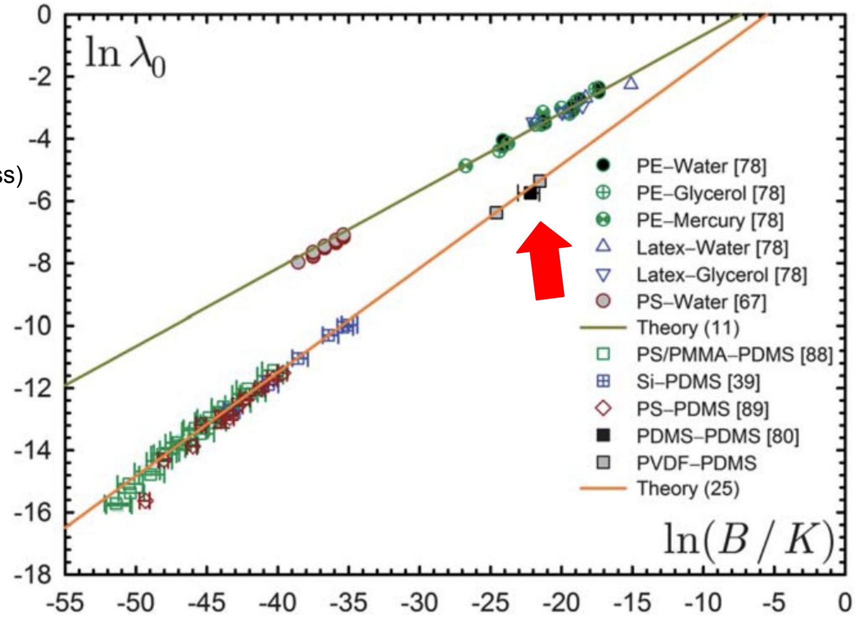


Fig. 3 Circular and triangular symbols correspond to data for liquid foundations from ref. 67 and 78 with $K = \rho g$. PE stands for polyester and PS stands for polystyrene. Square and diamond symbols correspond to data for elastic substrates from ref. 39, 88 and 89 with $K = E_s/3$. PMMA stands for polymethyl methacrylate and Si stands for silicon. Experiments using PVDF thin sheets of thickness 9 and 25 μm and partially cross-linked PDMS substrate have been performed to extend the spanned experimental domain ($E = 2.5 \pm 0.5$ GPa and $\sigma = 0.35$ for PVDF[90] and $E = 25 \pm 5$ kPa and $\sigma = 0.5$ for PDMS). The bending modulus B of polystyrene sheets used in ref. 67 has been computed using $E = 3 \pm 1$ GPa and $\sigma = 0.35$.[81,82] When not displayed, error bars have sizes similar to symbol sizes. SI units are used for λ_0 and the ratio B/K.

Calculation of B with Uncertainty

Given:

$$E = 25 \pm 5\,\text{kPa}$$
$$\sigma = 0.5\,\text{(for PDMS)}$$
$$\ln\left(\frac{B}{K}\right) = -22.25 \quad \text{(with uncertainty between -23.0 and -21.75)}$$

First, let's calculate K:

$$K = \frac{E}{3}$$

The values of E with uncertainty are:

$$E_{min} = 20\,\text{kPa}, \quad E_{max} = 30\,\text{kPa}$$

So, the corresponding values of K are:

$$K_{min} = \frac{20 \times 10^3}{3} \approx 6666.67\,\text{Pa}$$

$$K_{max} = \frac{30 \times 10^3}{3} = 10000\,\text{Pa}$$

Next, we calculate the central value of K:

$$K_{central} = \frac{25 \times 10^3}{3} \approx 8333.33\,\text{Pa}$$

Using the central value $\ln(B/K) = -22.25$:

$$\frac{B}{K_{central}} = e^{-22.25}$$

$$e^{-22.25} \approx 2.1623 \times 10^{-10}$$

So, the central value of B is:

$$B_{central} = K_{central} \times e^{-22.25} \approx 8333.33 \times 2.1623 \times 10^{-10} \approx 1.802 \times 10^{-6}\,\text{Pa}$$

Now, let's calculate the uncertainty in $\ln(B/K)$:

$$\Delta(\ln(B/K)) = \frac{-21.75 - (-23.0)}{2} = \frac{1.25}{2} = 0.625$$

The uncertainty in B due to $\ln(B/K)$ is:

$$\Delta B_{\ln(B/K)} = B_{central} \cdot \Delta(\ln(B/K)) = 1.802 \times 10^{-6} \cdot 0.625 \approx 1.126 \times 10^{-6}\,\text{Pa}$$

Additionally, the uncertainty in K is:

$$\Delta K = \frac{E_{max} - E_{min}}{3} = \frac{30 \times 10^3 - 20 \times 10^3}{3} = 3333.33\,\text{Pa}$$

$$\Delta B_K = e^{-22.25} \cdot \Delta K \approx 2.1623 \times 10^{-10} \cdot 3333.33 \approx 7.211 \times 10^{-7}\,\text{Pa}$$

Finally, we combine the uncertainties:

$$\Delta B_{total} = \sqrt{(\Delta B_{\ln(B/K)})^2 + (\Delta B_K)^2} = \sqrt{(1.126 \times 10^{-6})^2 + (7.211 \times 10^{-7})^2} \approx 1.335 \times 10^{-6}\,\text{Pa}$$

Thus, the final value of B with its uncertainty is:

$$B = (1.802 \pm 1.335) \times 10^{-6}\,\text{Pa}$$

Calculation of λ_0 with Uncertainty

Given:

$$E = 25 \pm 5\,\text{kPa}$$

$$B = 0.000001810366613 \pm 0.000000855156636\,\text{Pa} \quad (\text{for } \ln(B/K) = -22.25)$$

The central value of λ_0

$$\lambda_0 = 2\pi\left(\left(\frac{3B}{E}\right)^{1/3}\right) \times 100$$

Using the central values of B and E:

$$\lambda_0 = 2\pi\left(\left(\frac{3 \times 0.000001810366613}{25 \times 10^3}\right)^{1/3}\right) \times 100$$

$$\lambda_0 \approx 2\pi\left(2.172 \times 10^{-10}\right)^{1/3} \times 100$$

$$\lambda_0 \approx 2\pi \times 5.977 \times 10^{-4} \times 100$$

$$\lambda_0 \approx 0.38\,\text{cm}$$

Propagate the uncertainty in B and E

$$\frac{\partial\lambda_0}{\partial B} = \frac{2\pi}{(25 \times 10^3)^{2/3}}\left(\frac{3}{0.000001810366613}\right)^{2/3} \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial B} \approx \frac{2\pi}{1071.77} \times 1310 \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial B} \approx 0.0079\,\text{cm/Pa}$$

Propagate the uncertainty in B and E

$$\frac{\partial\lambda_0}{\partial B} = \frac{2\pi}{(25 \times 10^3)^{2/3}}\left(\frac{3}{0.000001810366613}\right)^{2/3} \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial B} \approx \frac{2\pi}{1071.77} \times 1310 \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial B} \approx 0.0079\,\text{cm/Pa}$$

$$\frac{\partial\lambda_0}{\partial E} = -\frac{2\pi(0.000001810366613)^{1/3}}{(25 \times 10^3)^{5/3}} \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial E} \approx -\frac{2\pi \times 0.0121}{2.915 \times 10^7} \times \frac{100}{3}$$

$$\frac{\partial\lambda_0}{\partial E} \approx -0.000027\,\text{cm/Pa}$$

The uncertainties in λ_0 are:

$$\Delta\lambda_0 = \sqrt{\left(\frac{\partial\lambda_0}{\partial B}\Delta B\right)^2 + \left(\frac{\partial\lambda_0}{\partial E}\Delta E\right)^2}$$

$$\Delta\lambda_0 = \sqrt{(0.0079 \times 0.000000855156636)^2 + (-0.000027 \times 5000)^2}$$

$$\Delta\lambda_0 \approx \sqrt{(6.75 \times 10^{-9})^2 + (0.135)^2}$$

$$\Delta\lambda_0 \approx 0.135\,\text{cm}$$

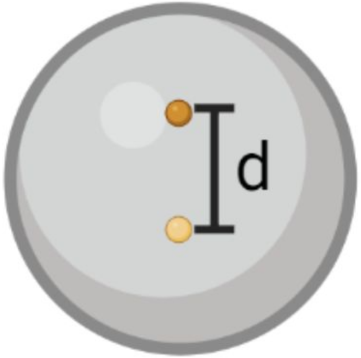
Final result:

$$\lambda_0 = 0.38 \pm 0.14\,\text{cm}$$

```python
import numpy as np
import matplotlib.pyplot as plt

def calculate_arc_length(amplitude, wavelength):
    x = np.linspace(0, wavelength, 1000)
    y = amplitude * np.sin(2 * np.pi * x / wavelength)
    dx = x[1] - x[0]
    dy = np.gradient(y, dx)
    ds = np.sqrt(1 + dy**2) * dx
    arc_length = np.sum(ds)
    return arc_length

def simulate_sinusoid_and_calculate(avg_distance_per_wrinkle, uncertainty,
label):
    wavelength = avg_distance_per_wrinkle  # distance between peaks
    amplitude = wavelength / 4  # amplitude is half the distance between a
peak and a valley (quarter of the wavelength)

    # Define the x values for one wavelength
    x = np.linspace(0, wavelength, 1000)  # simulate for one full wavelength

    # Define the sinusoidal function
    y = amplitude * np.sin(2 * np.pi * x / wavelength)

    # Calculate the arc length of the sinusoidal curve
    arc_length = calculate_arc_length(amplitude, wavelength)

    # Calculate the straight line length (horizontal distance)
    straight_length = x[-1] - x[0]

    # Calculate the extra length due to the curve
    extra_length = arc_length - straight_length

    # Print the results
    print(f"Data Set: {label}")
    print(f"Arc Length of the Sinusoidal Curve: {arc_length:.4f} cm")
    print(f"Straight Line Length: {straight_length:.4f} cm")
    print(f"Extra Length Due to the Curve: {extra_length:.4f} cm")
    print()

    # Plot the sinusoidal function
    plt.figure(figsize=(10, 4))
    plt.plot(x, y, label='Sinusoidal Curve')
    plt.hlines(0, x[0], x[-1], colors='r', linestyles='dashed', label='Straight Line')
    plt.xlabel('x (cm)')
    plt.ylabel('y (cm)')
    plt.title(f'Sinusoidal Function Simulation for One Wavelength ({label})')
    plt.legend()
    plt.grid(True)
    plt.show()

# Data Set 1
avg_distance_per_wrinkle_1 = 0.2471  # in cm
uncertainty_1 = 0.025  # in cm
simulate_sinusoid_and_calculate(avg_distance_per_wrinkle_1,
uncertainty_1, 'Data Set 1')

# Data Set 2
avg_distance_per_wrinkle_2 = 0.271  # in cm
uncertainty_2 = 0.015  # in cm
simulate_sinusoid_and_calculate(avg_distance_per_wrinkle_2,
uncertainty_2, 'Data Set 2')

# Data Set 3
avg_distance_per_wrinkle_3 = 0.2408  # in cm
uncertainty_3 = 0.014  # in cm
simulate_sinusoid_and_calculate(avg_distance_per_wrinkle_3,
uncertainty_3, 'Data Set 3')
```

Reducing the droplet size= the displacement of the catalyst is controlled by Brownian motion and gravitational force

Movement of the catalyst droplet



Brownian motion

$$D = \frac{k_B T}{6\pi\eta r}$$

$$Thermal\ Energy\ E_T = k_B T$$

D - diffusion coefficient
k_B - Boltzmann's constant
T - temperature
η - viscosity
r - particle size

Gravitational (Buoyancy) force

$$F = \rho V g$$

$$Potential\ Energy\ E_g = \Delta\rho V g d$$

d - Height of a particle traveled due to gravitational force
(Assume that d equals to r)

When $E_T > E_g$

$$\left(\frac{k_B T}{\Delta\rho g r^4} > 1\right)$$

Thermal energy counteracts gravitational potential energy

Critical radius (catalyst in PEG-400 at 20°C)

$$r_C \approx 1.3\ \mu m$$

Elastic Modulus (Young's Modulus): Measures the stiffness of a material in response to tensile or compressive stress.
Bending Modulus (Flexural Modulus): Measures the stiffness of a material in response to bending stress.

1. Geometric Considerations:
   - Young's modulus is a material property independent of the object's shape.
   - The bending modulus depends on the material's Young's modulus and the geometric properties of the object's cross-section.

Example

- Young's Modulus: If you pull on a steel rod, Young's modulus will tell you how much it will stretch.
- Bending Modulus: If you apply a force to the center of a steel beam supported at both ends, the bending modulus will tell you how much it will bend.