Membrane Case Study:

A Review of Sampling

Objective: Review the concept and methodology of component of variance studies with a case study.

Hunches and intuitive impressions are essential for getting the work started but it is only through the quality of numbers at the end that the truth can be told.

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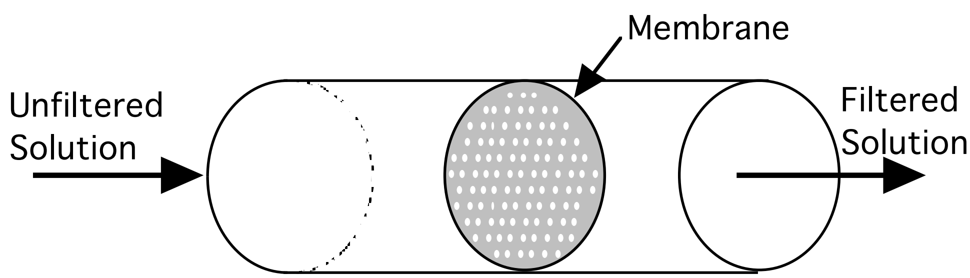
Product

Porous nitrocellulose membranes used in ultrafiltration devices.

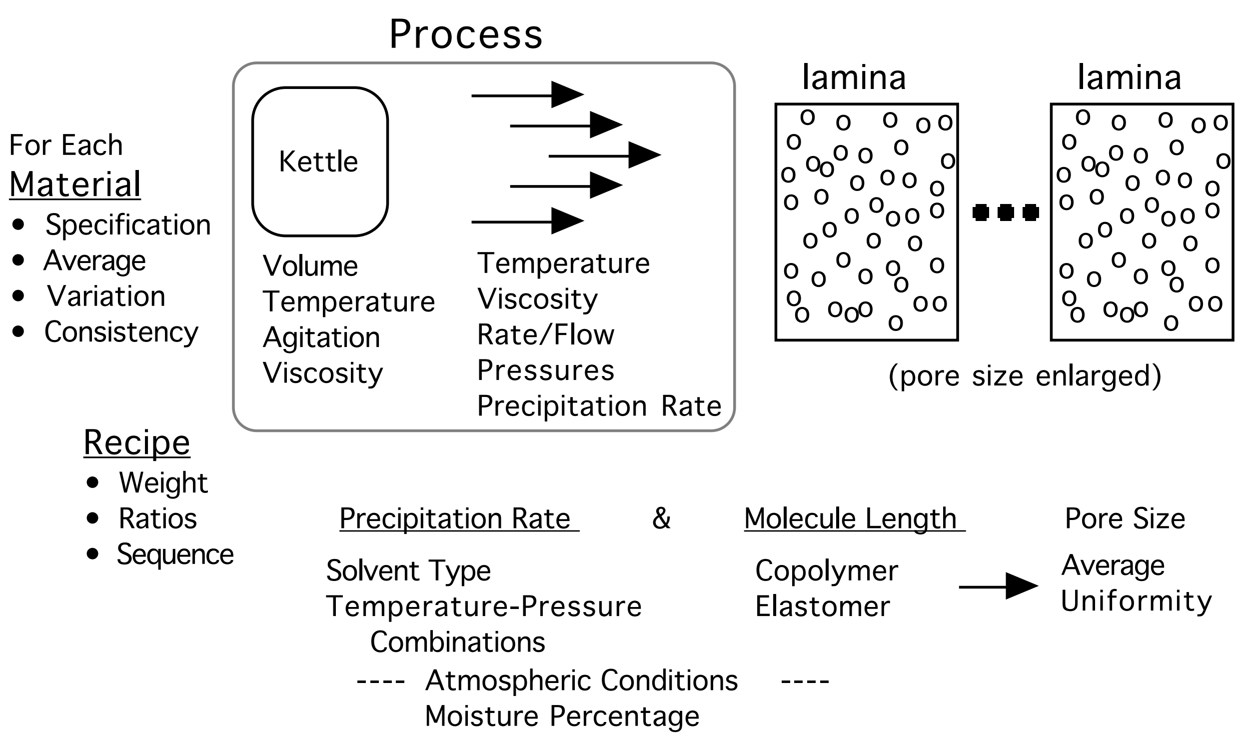


Pore size is a critical characteristic, receiving close and constant scrutiny from customers. Depending on the customers intended application, membranes with various pore sizes are required.

Average pore size, built to target, and uniformity in pore sizes are highly prized by customers who continuously identify new applications for the devices and who constantly find themselves having to meet more stringent requirements.

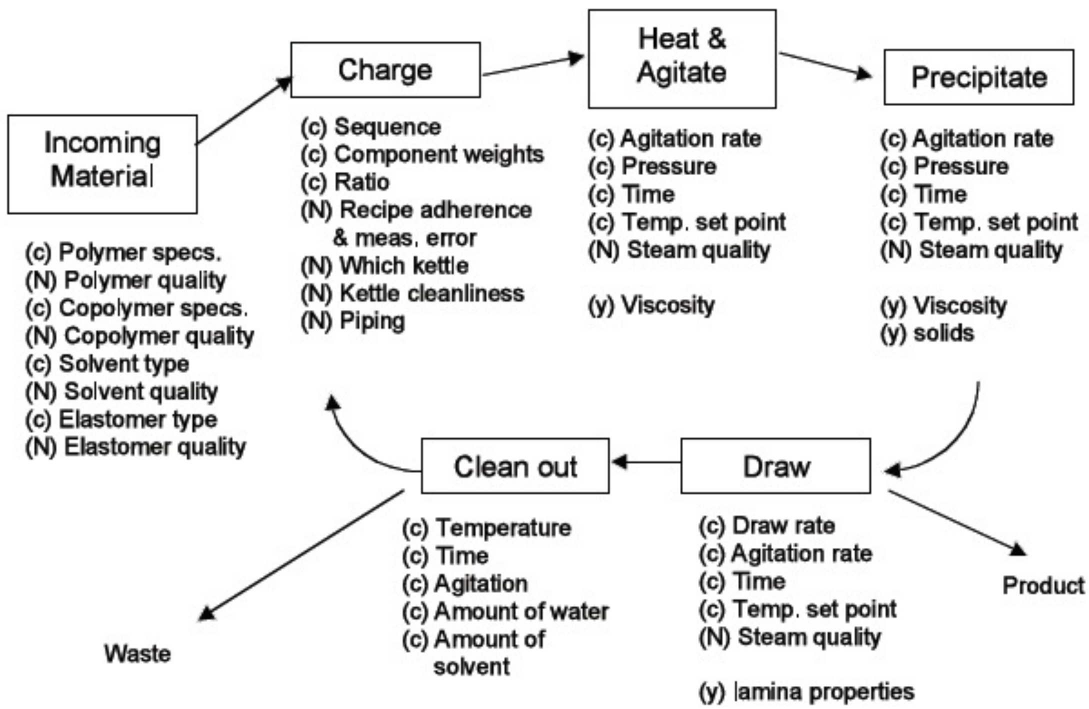
Process & Preliminary Map

Polymer is put into solution at an elevated temperature in this batch process. As temperature is reduced, polymer drops out of solution and forms a thin film membrane, sheets of which are called lamina.



Initial Work

The principle polymer is received in tubs from which many membrane laminas can be made. The supplier claims there is very little difference in raw polymer within a tub. A map for a potential DOE is created:



Objective & Questions

Discover how to reduce pore size variability (What factors affect this?). Where is the greatest source of variation in pore size?

- **the measurement system**,
- **the process**, or
- the **raw materials**?

Potential Sources Of Variation

Materials. Polymer chemistry, material properties, manufacturing variation and consistency, etc.

Process. Current equipment and technology, practices, and methods, and ability to adhere to product recipes and manufacturing instructions, ambient conditions, etc.

Measurement. Estimation of pore size, lighting, orientation of lamina, amount of magnification, etc.

Response (Y)

Each selected lamina is divided into a grid. One square is randomly chosen from the grid. The average pore diameter in this small area is then measured. Pore size is measured by taking a picture of the area (1000 times enlarged) and then estimating the average diameter of the pores in the enlarged picture using an optical comparator with a calibrated mylar overlay.

Strategy (Predicted next steps)

In order to evaluate the various sources, the manufacturer will continue to study:

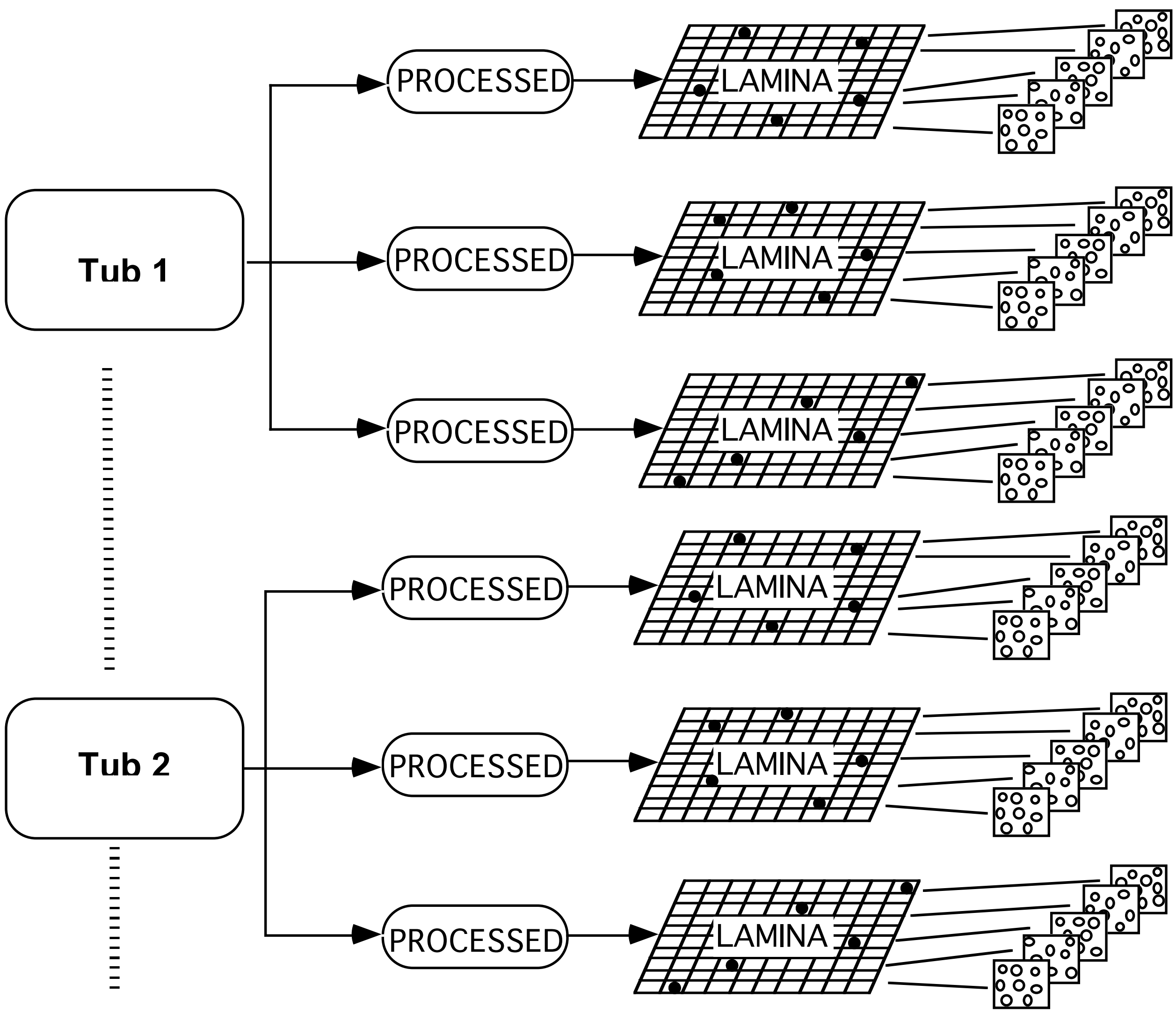
- **measurement system…**if within lamina ("instantaneous") variation is large.
- **the manufacturing process…** if between lamina, within material lot, ("short term") variation is large.
- **raw materials…** between tub ("long term") variation is large.

Initial Proposed Sampling Plan:

1. Sample three lamina from the same tub (L).

2. For each lamina, measure a random sample of 5 squares (M).

3. Repeat this for 10 tubs (T).



Assignment:

1. Draw a tree for the initial proposed sampling plan.

2. Draw, compare and contrast the trees describing the sampling scheme where[1]:
   - 5 tubs are selected. Three batches are sampled from each tub. Two lamina are sampled from each batch. One square from each lamina, each "square" is measured twice. Identify where potential sources of variation are captured on your tree. These should include between tub, within tub, between batch, within batch, lamina-to-lamina, within lamina and measurement error.
   - 7 tubs are selected. Only one batch is sampled from each tub. Two lamina from each batch are measured, one square from each. Identify where potential sources of variation are captured on your tree. These should include between tub, within tub, between batch, within batch, lamina-to-lamina, within lamina and measurement error.

[1] All plans use the same Y (a random sample of one square selected from a grid).

- 2 tubs are selected. For every tub, two batches are sampled. Two lamina per batch are collected. From each lamina, two squares are selected and they are measured twice. Identify where potential sources of variation are captured on your tree. These should include between tub, within tub, between batch, within batch, lamina-to-lamina, within lamina and measurement error.

3. Using the initial proposed plan:

    a. Analyze the data. Assess stability and if appropriate calculate estimates of the components of variation.

    b. Answer the questions that follow the data.

Original Data, Averages and Ranges

Tub	Lamina	Measurements, Y_i 1	2	3	4	5	$\overline{Y}_L$	R_{WL}
1	1	0.504	0.649	0.582	0.603	0.673	0.6022	0.169
1	2	0.509	0.669	0.605	0.531	0.561	0.5750	0.160
1	3	0.628	0.629	0.570	0.455	0.553	0.5670	0.174
2	4	0.521	0.563	0.532	0.560	0.626	0.5604	0.105
2	5	0.527	0.600	0.581	0.484	0.622	0.5628	0.138
2	6	0.537	0.483	0.503	0.521	0.447	0.4982	0.090
3	7	0.600	0.537	0.598	0.653	0.571	0.5918	0.116
3	8	0.646	0.545	0.528	0.622	0.606	0.5894	0.118
3	9	0.490	0.542	0.599	0.622	0.508	0.5522	0.132
4	10	0.615	0.530	0.651	0.549	0.614	0.5918	0.121
4	11	0.555	0.501	0.492	0.507	0.391	0.4892	0.164
4	12	0.537	0.565	0.552	0.459	0.442	0.5110	0.123
5	13	0.611	0.587	0.465	0.663	0.482	0.5616	0.198
5	14	0.465	0.465	0.518	0.527	0.566	0.5082	0.101
5	15	0.599	0.608	0.634	0.585	0.542	0.5936	0.092
6	16	0.498	0.591	0.511	0.532	0.441	0.5146	0.150
6	17	0.462	0.464	0.531	0.480	0.389	0.4652	0.142
6	18	0.349	0.569	0.525	0.553	0.510	0.5012	0.220
7	19	0.603	0.489	0.487	0.596	0.568	0.5486	0.116
7	20	0.616	0.578	0.596	0.613	0.623	0.6052	0.045
7	21	0.709	0.549	0.526	0.587	0.535	0.5812	0.183
8	22	0.506	0.514	0.462	0.554	0.529	0.5130	0.092
8	23	0.513	0.611	0.514	0.490	0.510	0.5276	0.121
8	24	0.613	0.553	0.544	0.534	0.548	0.5584	0.079
9	25	0.630	0.606	0.575	0.646	0.609	0.6132	0.071
9	26	0.522	0.654	0.573	0.567	0.532	0.5696	0.132
9	27	0.697	0.631	0.601	0.640	0.615	0.6368	0.096
10	28	0.522	0.555	0.542	0.571	0.605	0.5590	0.083
10	29	0.543	0.583	0.514	0.565	0.642	0.5694	0.128
10	30	0.621	0.600	0.632	0.604	0.677	0.6268	0.077

Questions to be Answered with the Initial Proposed Plan

1. a. What is the average range for within lamina variation in pore size?
   b. Is the pore variation consistent?
   c. What is the average pore size for all lamina?
   d. What is the estimated variance of the within lamina pore size?

2. a. Is lamina variation consistent across all tubs?
   b. What is the estimated variance lamina-to-lamina?

3. a. Is the tub-to-tub variation predictable?
   b. What is the estimated variance from tub-to-tub in pore size?
   c. What is the average pore size for all tubs included in this study?

4. a. What is the total estimated variance in pore size?
   b. What is the percent contribution of:
      - Pore
      - Lamina
      - Tub

      to the total estimated variance?

5. Do these estimates of components of variance really estimate the 'true' components of variation? (In practice, how would you know how 'good' the estimates are?)

Notes: