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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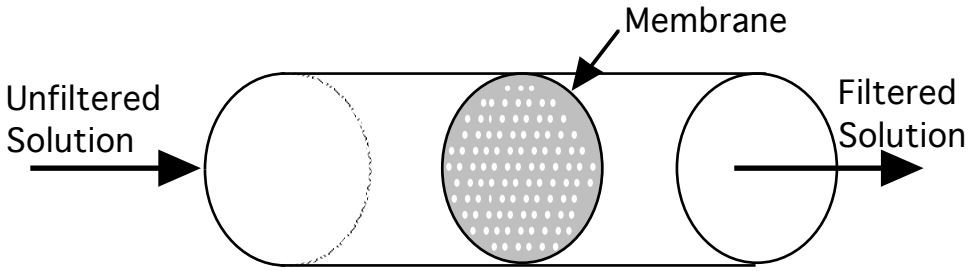
Product

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 uniform pore sizes are highly prized by customers who annually identify new applications for the devices and who constantly find themselves having to meet more stringent requirements.

The current work will be focused on pore size uniformity. Other properties that are important to the customer are the following:

- Hydrophobic properties (ability to repel water and absorb chemicals dissolved in water; e.g., desalination.)

- Hydrophilic properties (ability to absorb water and leave a substrate behind, e.g., removing water from a pharmaceutical products)

- Pressure (ability to withstand high pressures, e.g., high pressure filtration)

- Food and Drug Administration (ability to withstand autoclaving, live steam, ethylene oxide or radiation, e.g., FDA-monitored micro biological or pharmaceutical work)

- Cost

Also important to competitive position are the following:

- Availability and cost of polymer, copolymer, elastomer and solvent
- Efficiency of solvent recovery
- Throughput
- Ability of membranes to absorb charged chemical agents (enhances filtration ability, especially with proteins)

Background

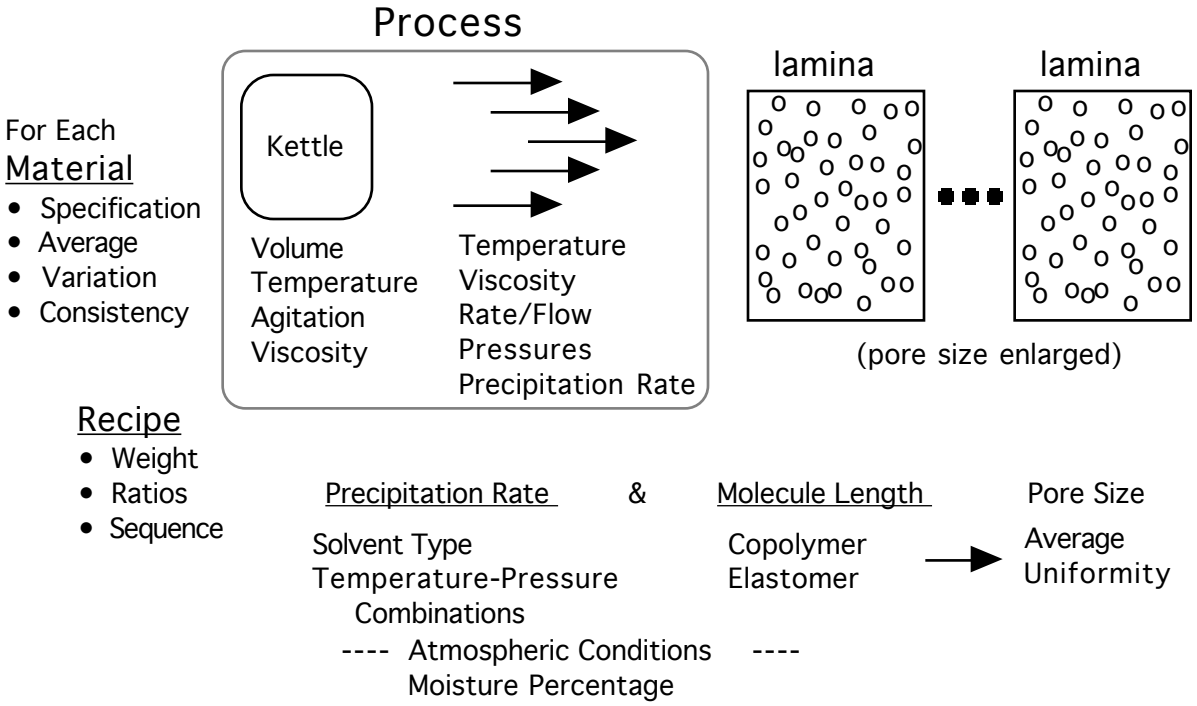
Over time and across many runs and much production, **average** porosity is consistently on target.

However, several customers have indicated that more uniform porosity will be required in the near future.

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 which are called lamina.



Initial Work

The principle polymer is received in rail cars from which many membrane laminas can be made.

The engineering team assumes that there is very little difference in raw polymer within a car. (Supplier data).

Other materials are received and are used at different rates.

Objective & Questions

Discover how to reduce pore size variability.

Where is the greatest source of variation in pore size?

- in **the process**,
- in the **repeatability of the process**, or
- in the **raw materials**.

Potential Sources Of Variation

Materials. Their properties, variations, and consistency.

Process. Includes current equipment and technology, practices, and methods, and best current knowledge as represented in product recipes and manufacturing instructions. The COV will be conducted in 'blind' fashion. (Why?)

Response

Each selected lamina is divided into a grid. Five squares are randomly chosen from the grid. The average pore diameter in this small area is then measured. Pore size can be measured by taking a picture of the area (1000 times enlarged) and then estimating the average diameter of the pores in the enlarged picture. The measurement process has "passed" multiple MSEs over time.

Strategy

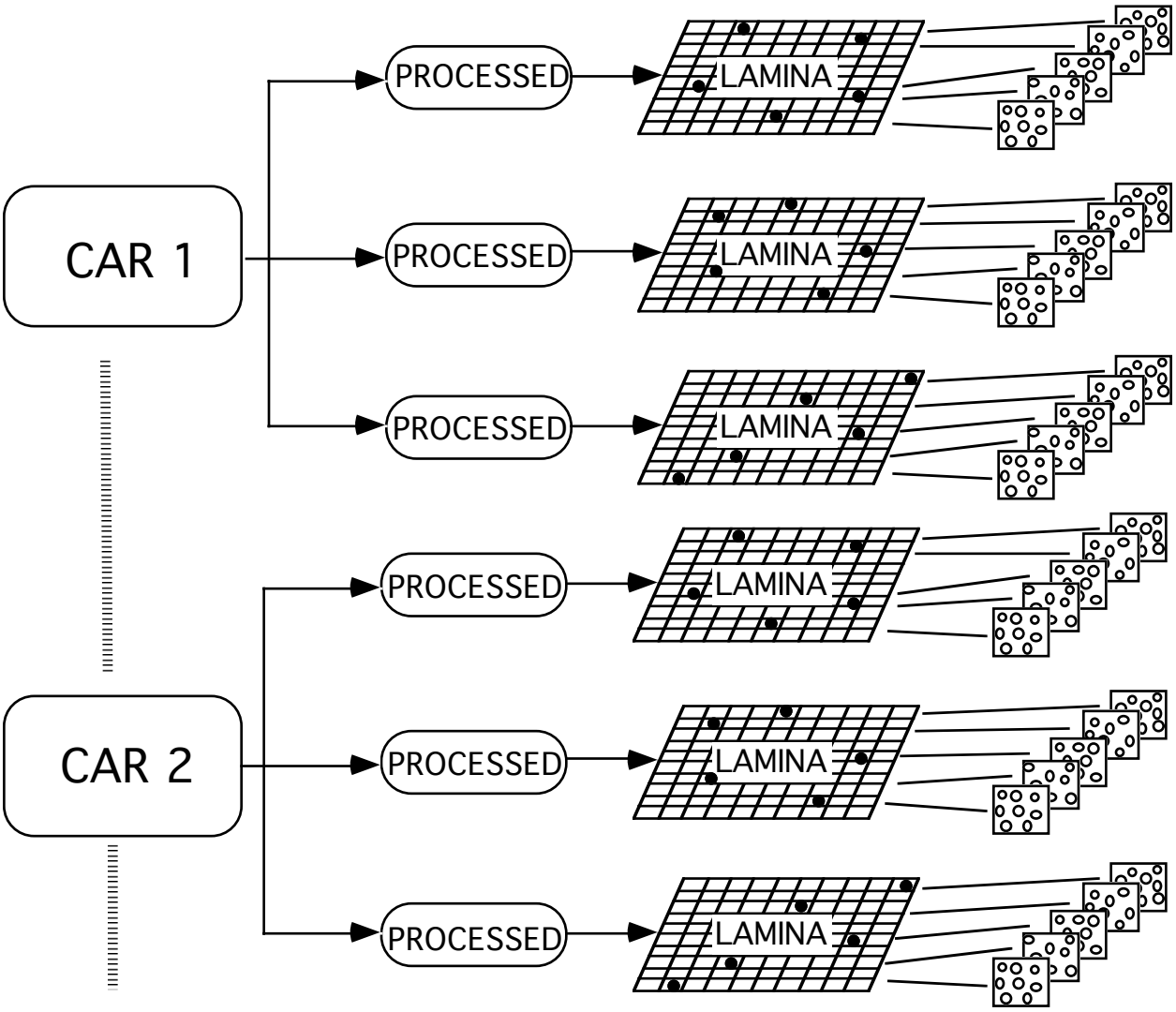
In order to evaluate the various sources, the manufacturer will sample:

- **process** if within lamina ("instantaneous") variation is large.
- **repeatability of the process** if between lamina, but within a material lot, ("short term") variation is large.
- **raw materials** between car ("long term") variation is large.

Notes:

Initial Proposed Sampling Plan:

1. Sample three lamina from the same car.
2. For each lamina, measure a sample of 5 pores.
3. Repeat this for 10 cars.



Assignment:

Assignment:

1. Draw a tree for the proposed sampling plan.

2. Draw alternative trees describing the sampling scheme where:
   - Multiple batches are produced from each car and three batches are sampled from each car. One lamina is sampled from each batch. (Multiple lamina are produced from each batch). Identify where potential sources of variation are captured on your tree. These should include between rail car, within rail car, between batch, within batch, and measurement error.
   - Only one batch is produced from each car. Each batch is measured twice. Identify where potential sources of variation are captured on your tree. These should include between rail car, within rail car, between batch, within batch, and measurement error.

- Multiple batches are produced from each car, but each car provides material to three lines. For every rail car three batches are sampled; one batch for each line. Each sample is measured twice. Identify where potential sources of variation are captured on your tree. These should include between rail car, within rail car, between line, within line, between batch, within batch, and measurement error.

Using the proposed plan:

1. Analyze the data with control charts. Assess stability and if appropriate calculate estimates of the components of variation.

2. Answer the questions that follow the data.

Original Data, Averages and Ranges

Car	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 this Plan

1. a. What is the average value for within lamina variation in pore size?
   b. Is within lamina variation consistent?
   c. What is the average pore size for all lamina?

2. a. What is the average within car variation, lamina to lamina?
   b. Is within car variation consistent across all cars?

3. a. What is the average deviation from car to car in pore size?
   b. Is this car to car variation predictable?
   c. What is the average pore size for all cars included in this study?

4. a. What is the total variance in pore size?

   b. What is the contribution of:

   - within lamina variance to the total variance?
   - within car variance to the total?
   - car to car differences?

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