Emulsion Processing
- Homogenization -

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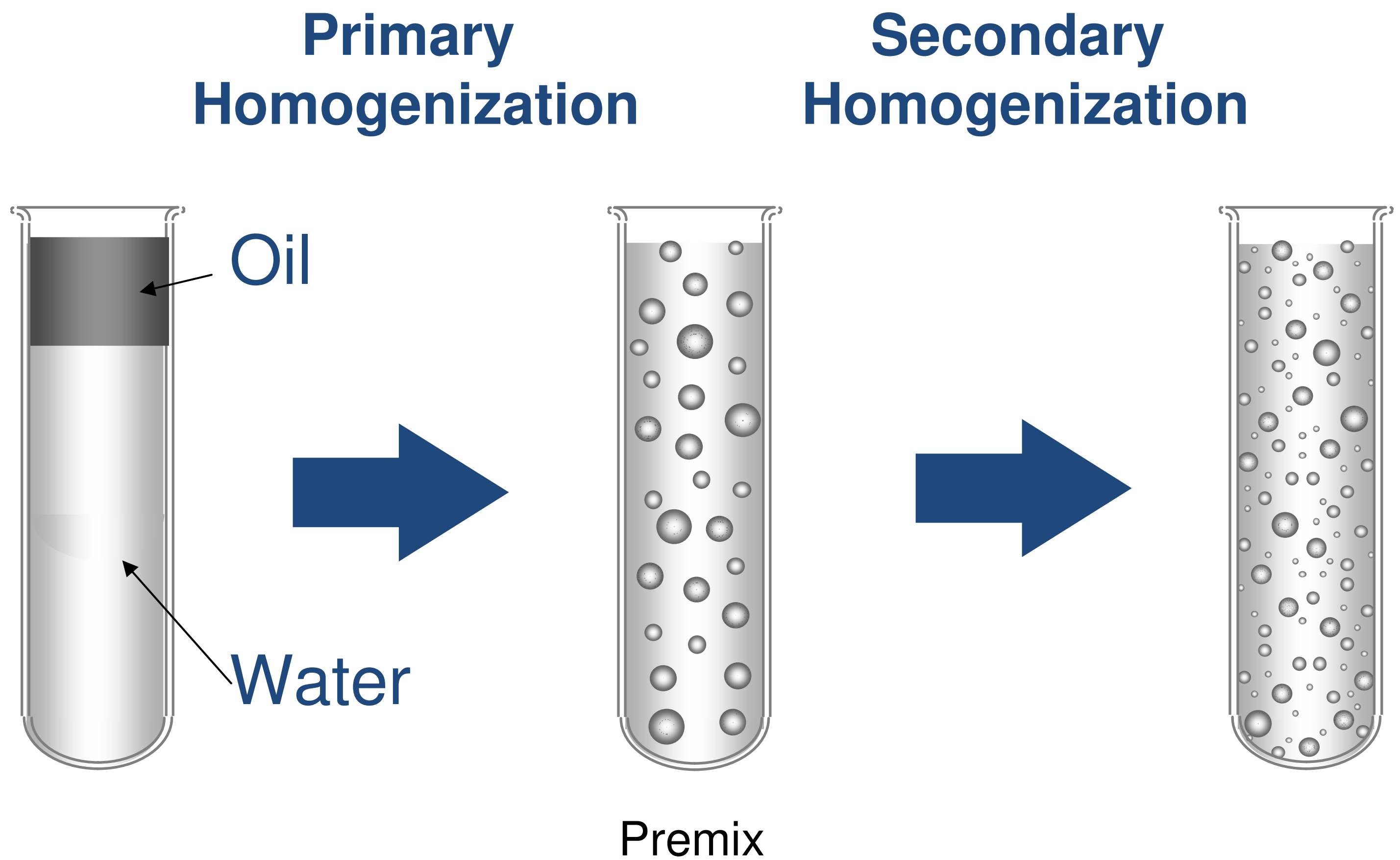
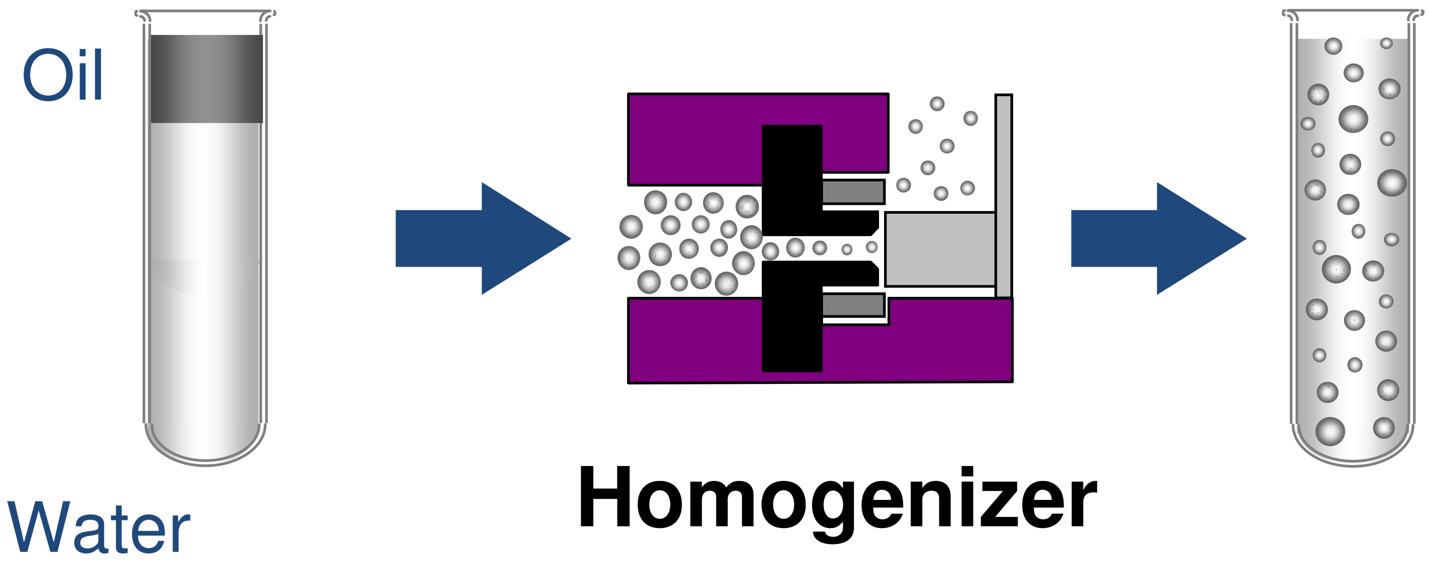
Emulsion Workshop

November 13-14th, 2008, Amherst, MA

Principle of Emulsion Formation

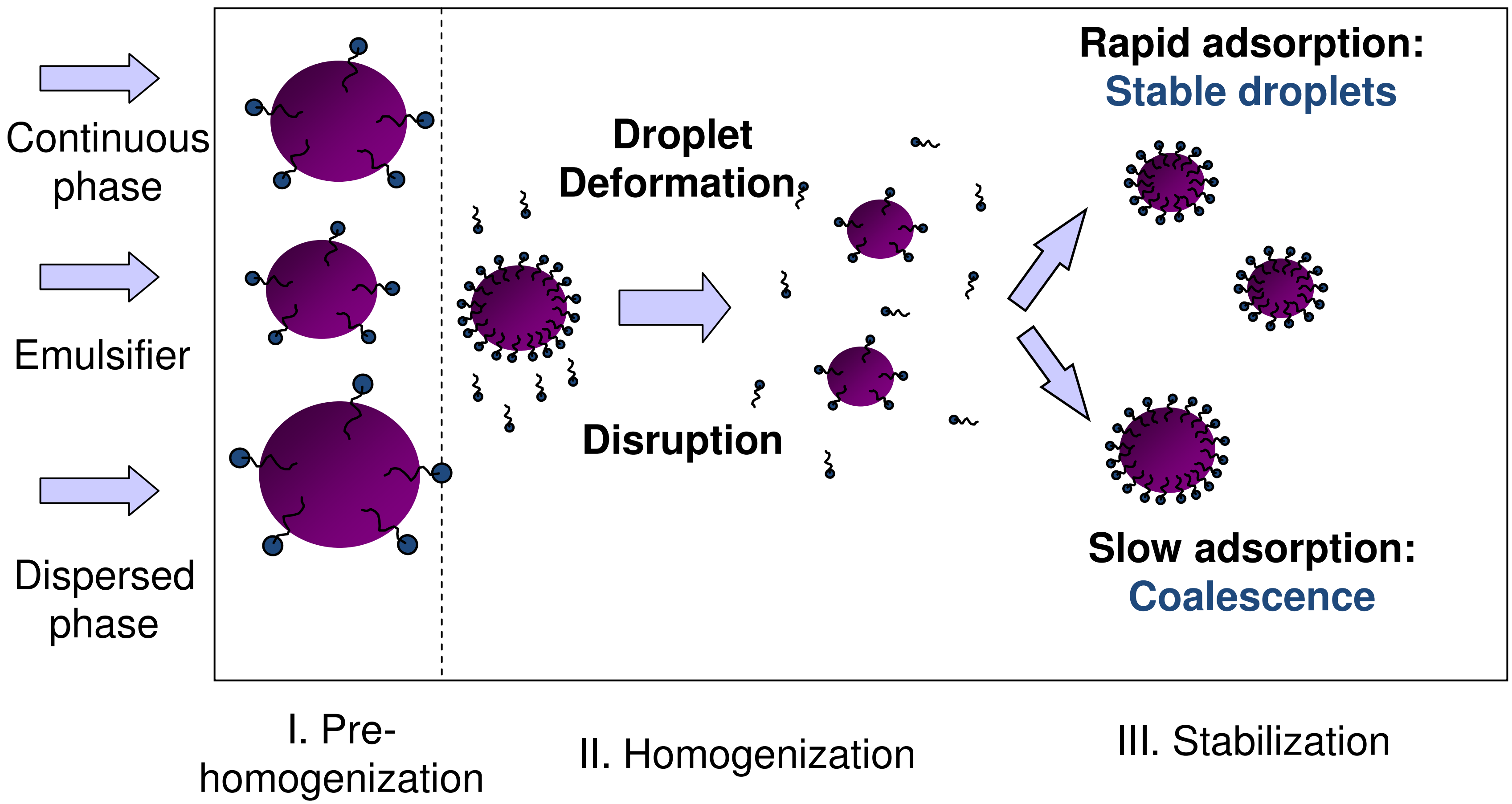


Emulsion Processing: Homogenizers

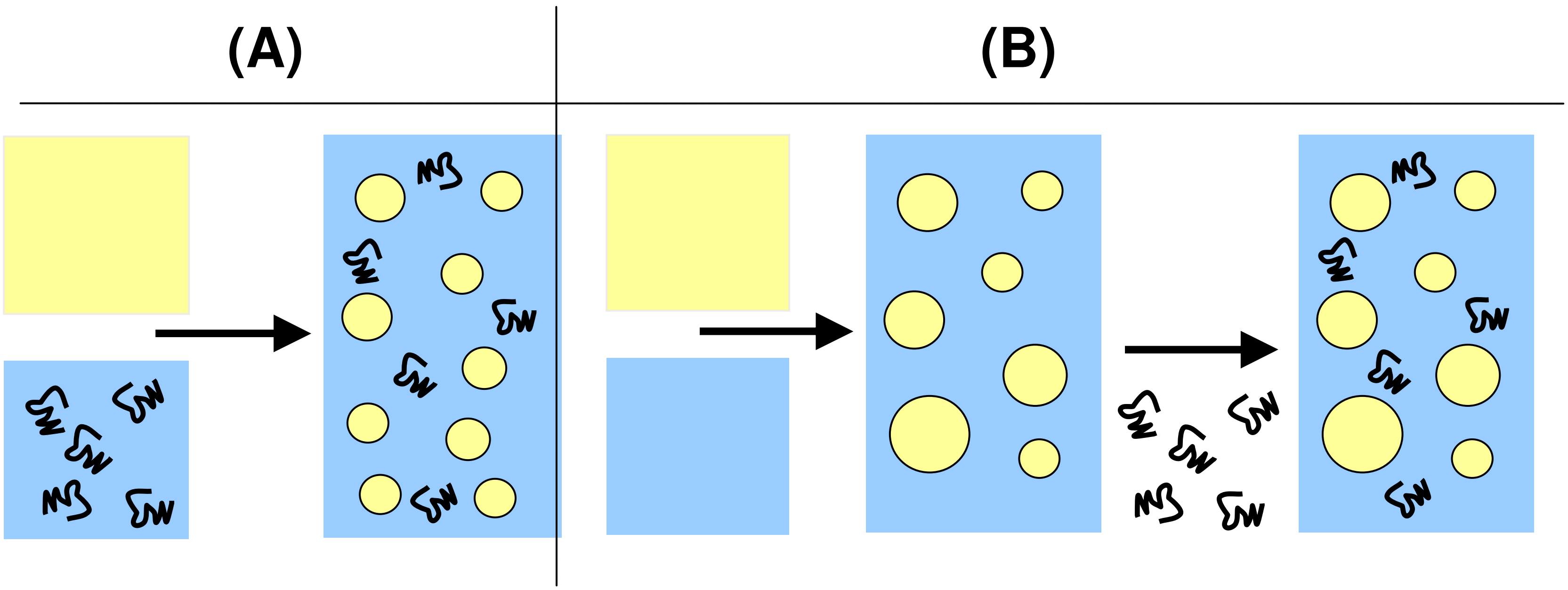


Homogenization is a unit operation using a class of processing equipment referred to as ***homogenizers*** that are geared towards reducing the size of droplets in liquid-liquid dispersions

Physiochemical Processes Occurring During Homogenization



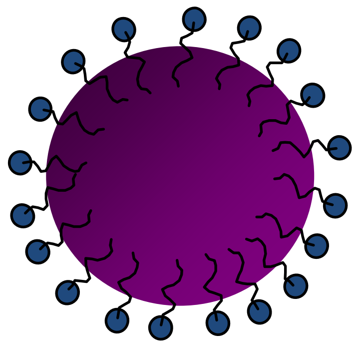
General Homogenization Options



The order of ingredient addition and homogenization may have a large impact on product properties

Homogenization: Process Parameters

- **Energy density**
  - minimum droplet size achievable
- **Energy efficiency**
  - heat losses - manufacturing costs
- **Volume Flow Rates**
  - throughput - production time
- **Product rheology**
  - limitations - materials that can be homogenized



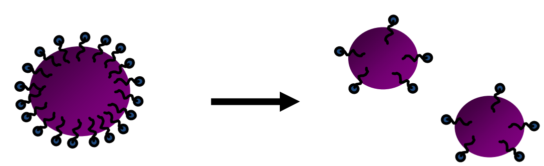
The Physics of Droplet Disruption

- **Maintaining Force.** Drop shape maintenance forces (Laplace Pressure)
- **Disruptive Force.** Drop disruption is due to drop-surface applied tangential stresses
- **Weber-Number (We):** The ratio between drop disrupting and drop maintaining forces, drop disruption occurs only above a critical Weber number
  - $We < We_{crit}$ or $t_{br} < t_{br,crit}$ → droplet deformation
  - $We > We_{crit}$ or $t_{br} > t_{br,crit}$ → droplet disruption
- **Deformation Time.** Droplets must be exposed to tangential stresses for a sufficient amount of time

$$p_c = \gamma\left(\frac{1}{r_1} + \frac{1}{r_2}\right) = \gamma\frac{4}{d}$$

$$We = \frac{\tau}{p_c} = \frac{\tau d}{4\gamma}$$

$$t_{br,crit} = \frac{\eta_d}{\tau - p_c}$$



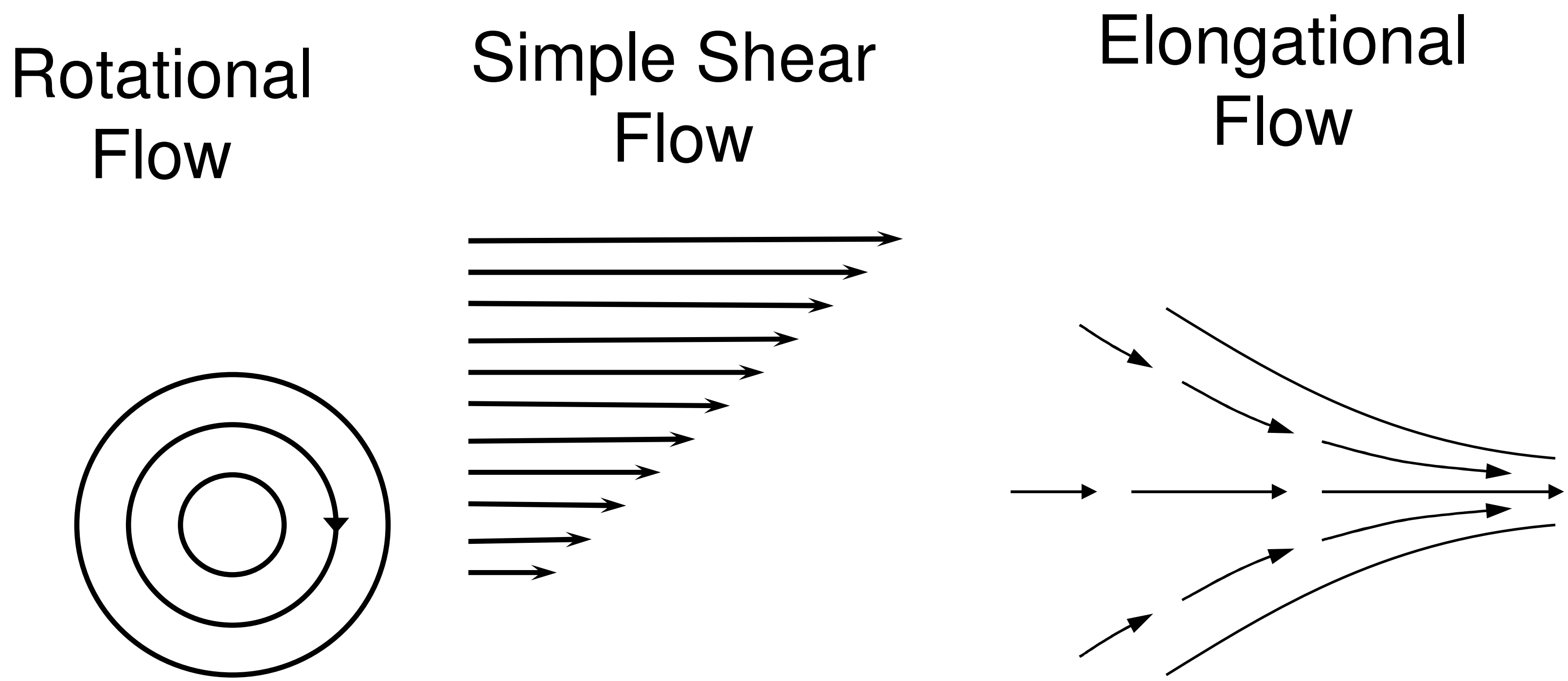
Key Parameter: Energy Density E_v

$$E_v = \frac{\text{Energy input } E}{\text{homogenized volume } V} = \frac{P}{\dot{V}} = P_v t_v$$

- The volume specific energy input or the energy density E_v can simply be calculated from the power consumption and the volume flow rate
- The mean droplet diameter may often be empirically related to the energy density, *IF*, all other parameters are kept constant!

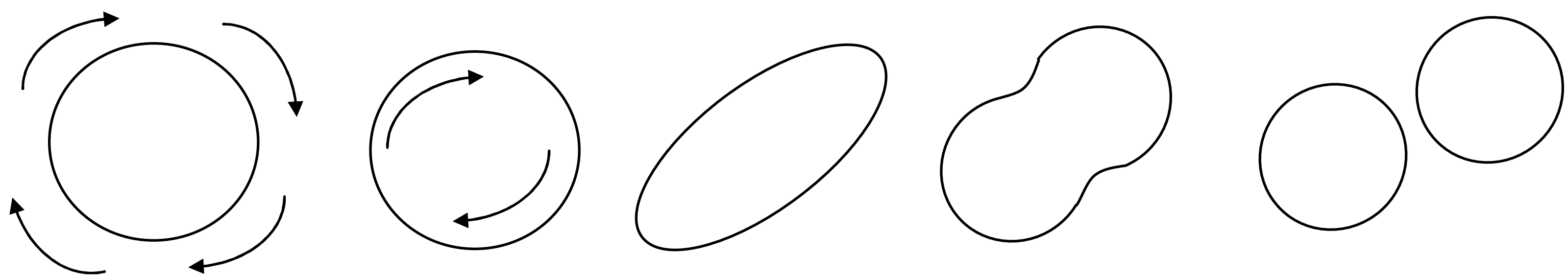
$$x_{1,2} = C\left(E_v\right)^b$$

How Droplets Are Disrupted: Flow Situations in Homogenizers



- Flow profiles are complex based on geometry of homogenizer.
- Flow may be laminar (rotational, simple shear, elongation) or turbulent.

Simple Deformation Scenarios For Liquid-Liquid Dispersions



Rotation of whole droplet

Circulation of fluid within droplet

Elongation of droplet

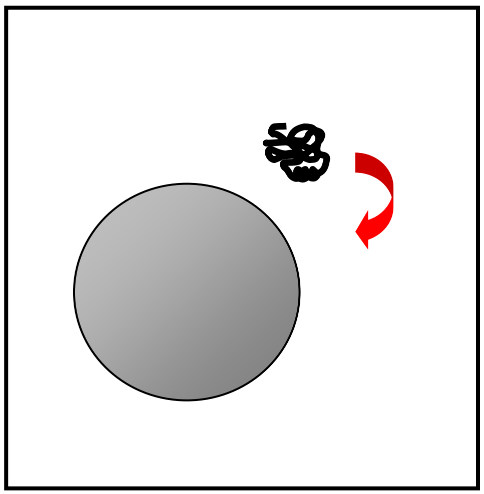
“Neck” Formation

Disruption of droplet

Increased Exertion of Stress Due to Superimposed Flow Profile

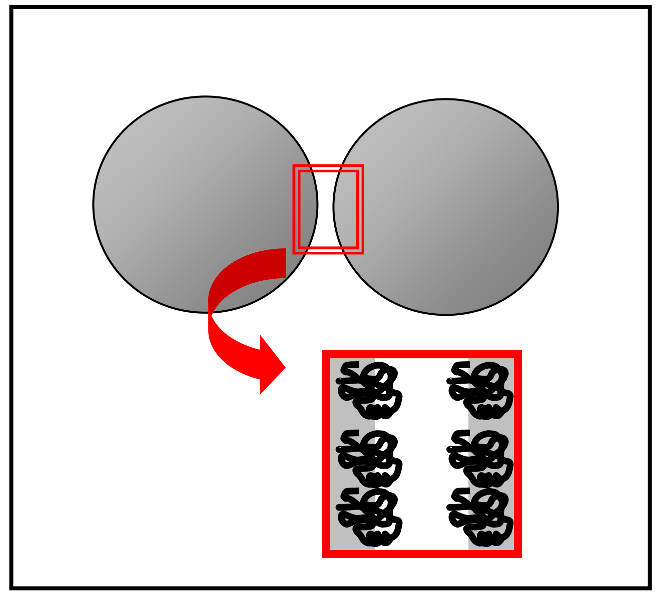
Role of Emulsifiers in Emulsion Formation and Stabilization

Formation



- Rapidly adsorb
- Lower interfacial tension
- Facilitate breakup

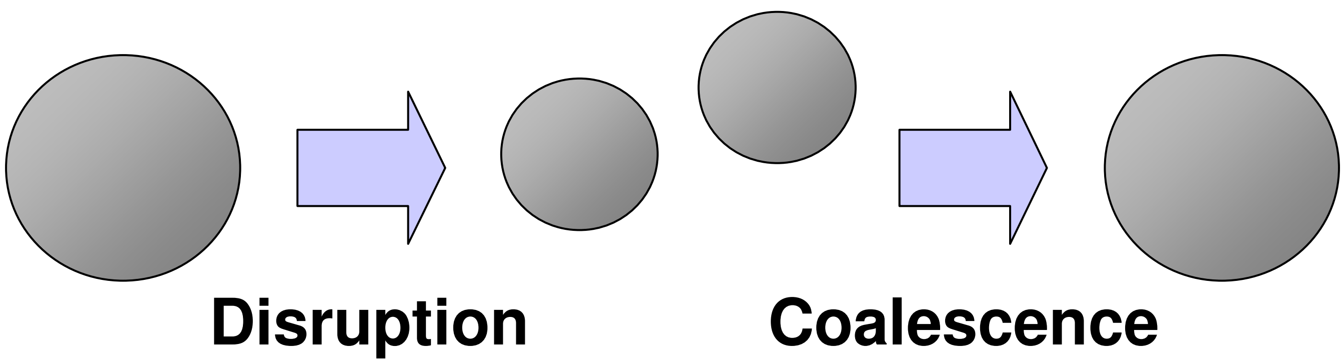
Stabilization



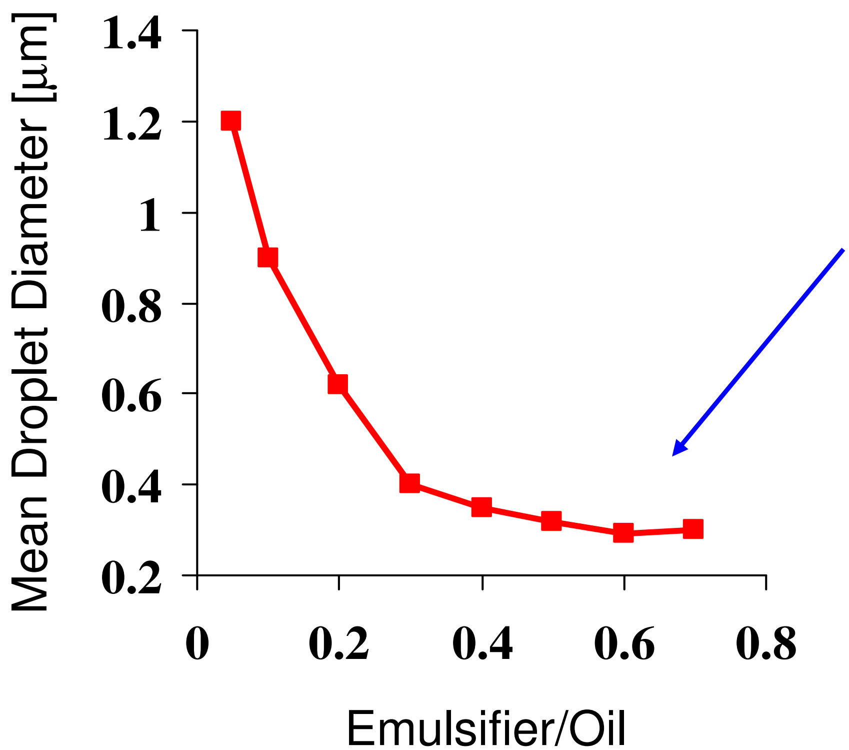
- Generate repulsive forces
- Form resistant membrane
- Prevent Coalescence

Optimum Emulsifier Characteristics for Emulsion Formation

- Objective: to generate small stable droplets
  - Rapid adsorption
  - Lower interfacial Tension
  - Form protective membrane



Factors Affecting Droplet Size: Emulsifier Concentration

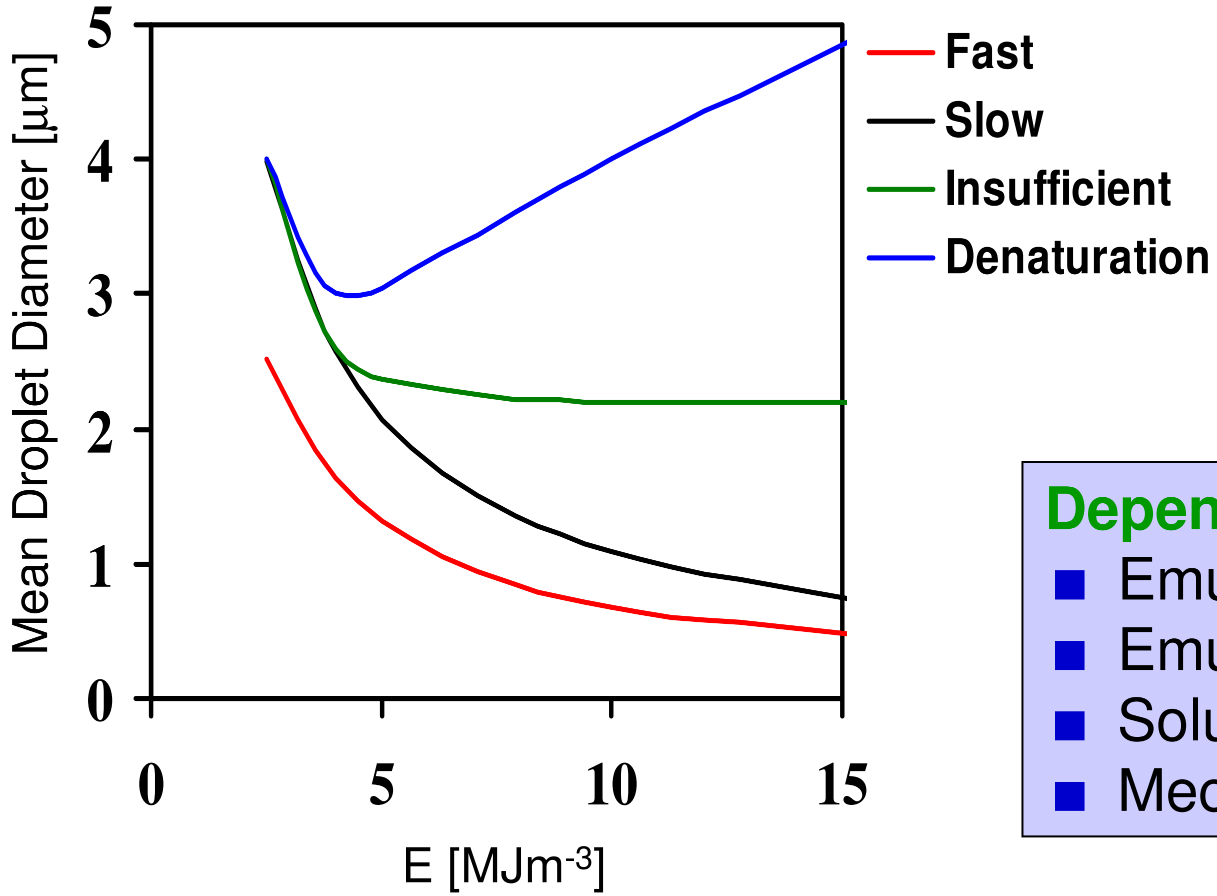


Once the emulsifier concentration exceeds a certain level the droplet size depends on the energy input of the homogenizer.

Fixed Homogenization Conditions (1000 psi)

Corn O/W emulsion - Pandolfe (1995)

Factors Affecting Droplet Size: Emulsifier Type



Depends on:

- Emulsifier Type
- Emulsifier Conc.
- Solution Conditions
- Mechanical Device

Characterizing Emulsifier Efficiency During Homogenization

- Maximum amount of oil that can be homogenized by fixed amount of emulsifier using standardized conditions (homogenization, pH, I, T)
  - EAI (g oil / g emulsifier) – Emulsifier activity index
- Minimum amount of emulsifier required to achieve a given droplet size using standardized conditions (ϕ, homogenization, pH, I, T)
  - c_{min} (g emulsifier / g oil)
- Minimum droplet size that can be achieved by homogenization
  - r_{min} (μm)

Homogenizers

High Speed Blender

High Pressure Homogenizers

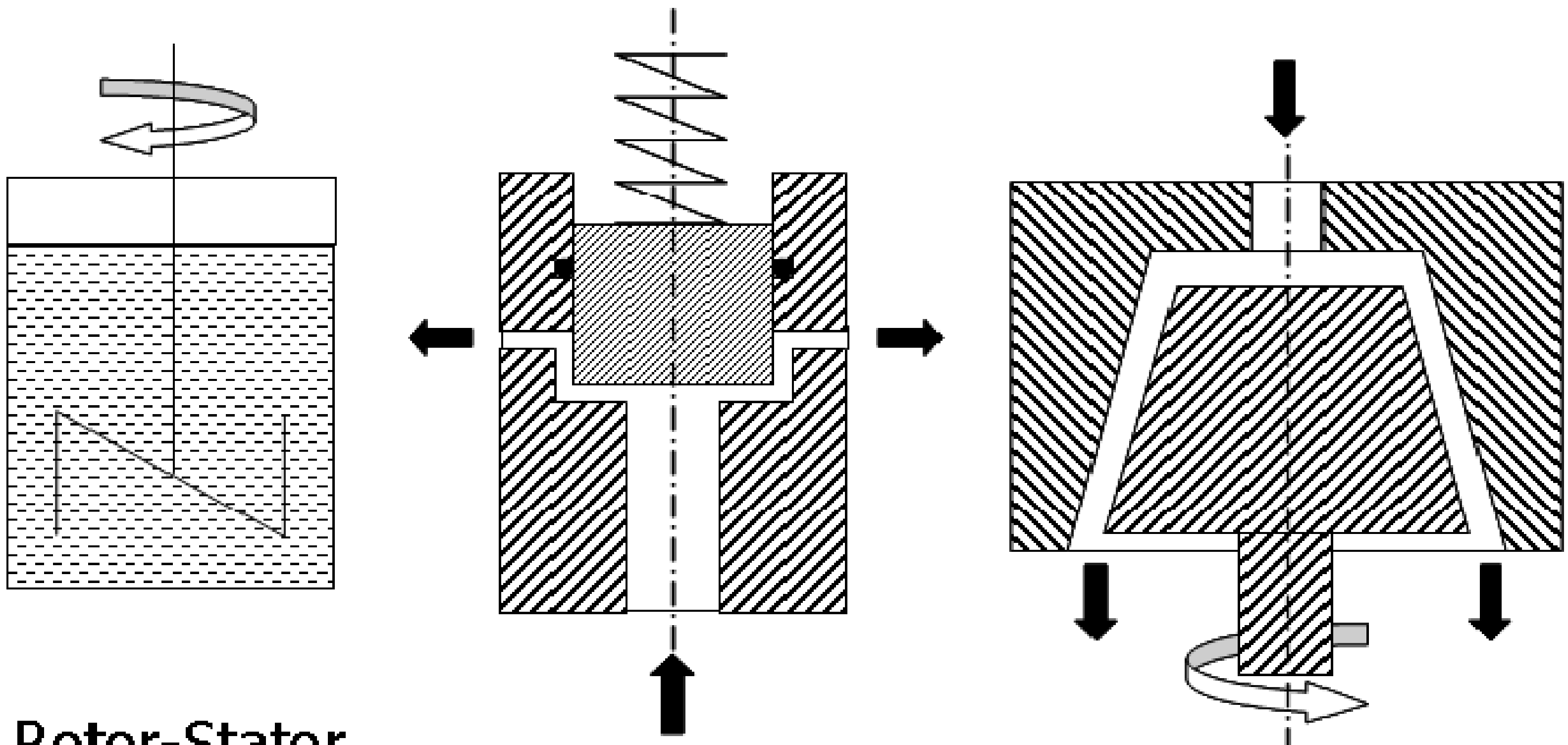
Colloid Mill

High Shear Dispersers

Ultrasonic Disruptor

Membrane Homogenizers

Homogenizers: A General Overview (I)

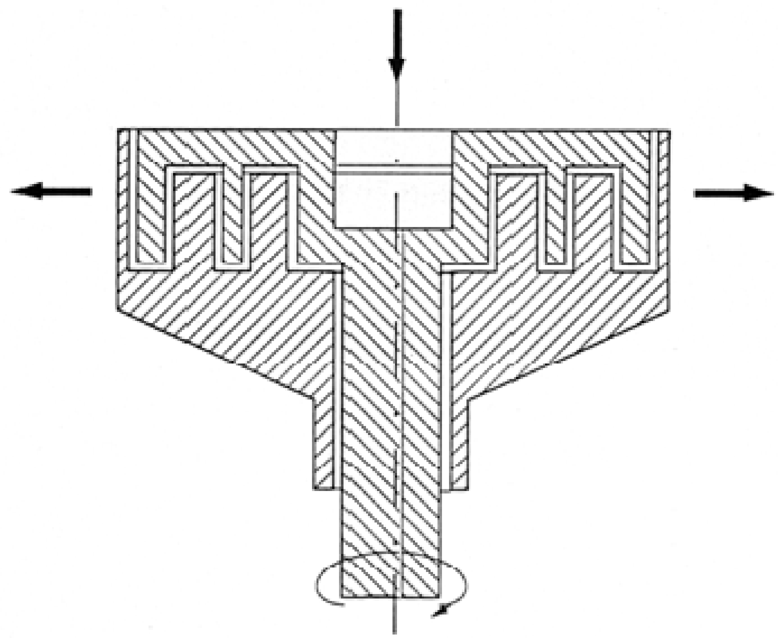


Rotor-Stator Systems (Blenders)

High-Pressure Homogenizers / Microfluidizer

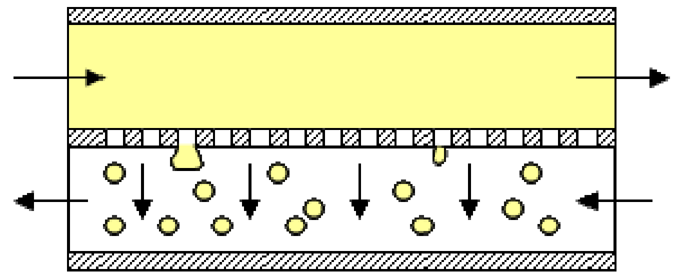
Colloid Mill

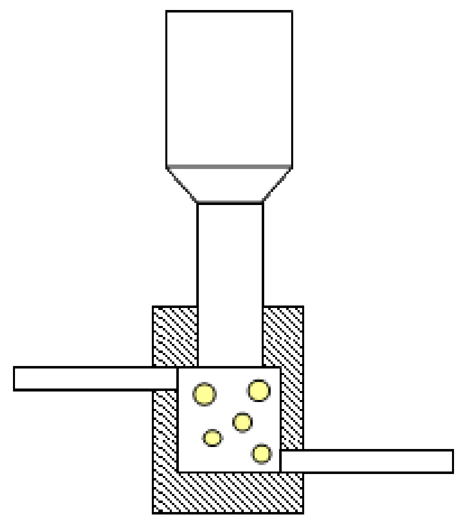
Homogenizers:
A General Overview (II)



High Shear Disperser
(Turrax)

Membrane Homogenizer

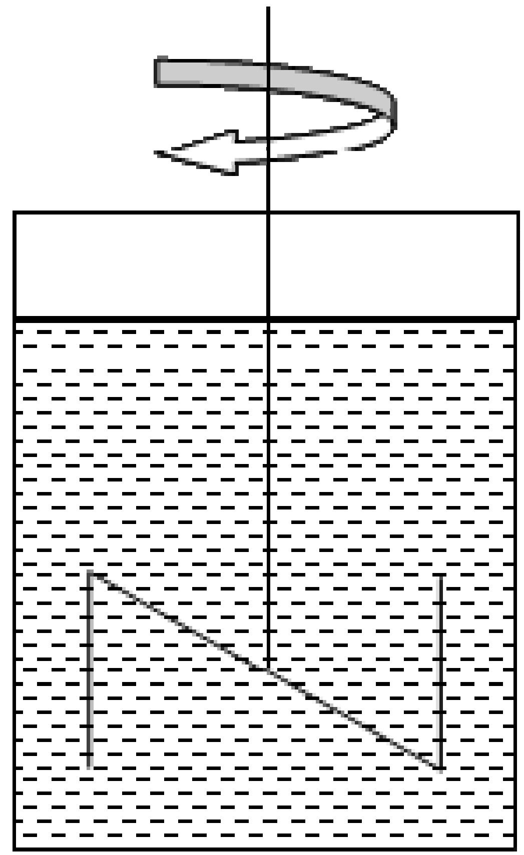




Ultrasonicator

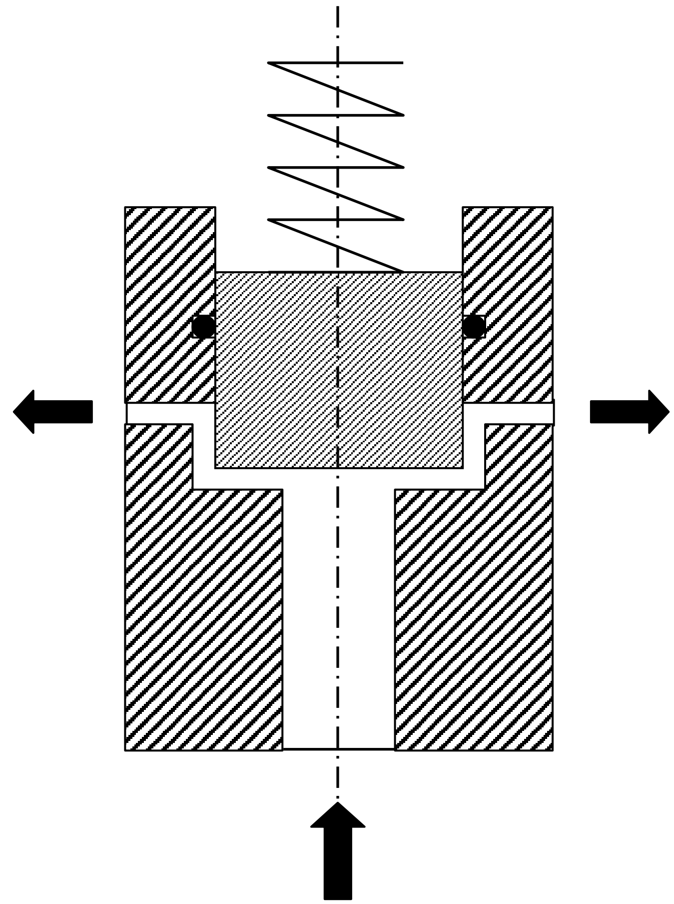
I. High Speed Blenders

- Low volume specific energy input (energy density)
- Energy input highly distributed in the stirred vessel (regions of low and high shear)
- Blender geometry and rotational speed are the prime parameters → turbulent flows preferred.
- Broad particle size distribution, large particles
- Need to avoid air incorporation to avoid foam formation
- Fairly inexpensive usually used for premix production



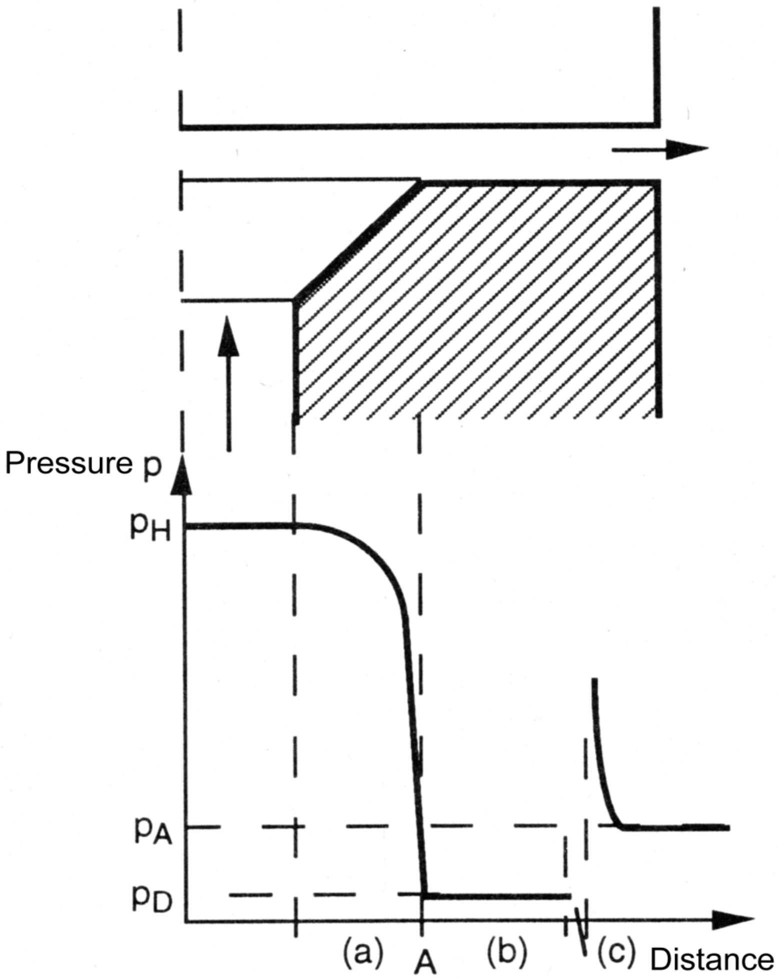
II. High Pressure Homogenizer (HPH)

- Most common used homogenizer in the food industry (milk, cream etc.)
- Disruptive energy comes from relaxation of high pressure build up across homogenization valve
- Pressures typically range from 50 to 500 bar (microfluidizer up to 1600 bar)
- Homogenization valve geometry of key importance → influences flow profile
- Homogenization may be single or multiple stage



Shear, Turbulent & Cavitation Forces

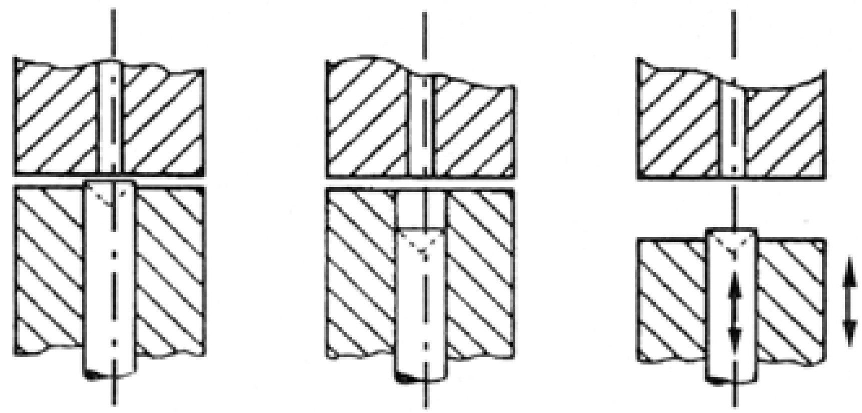
Physical Process Inside the Homogenization Valve

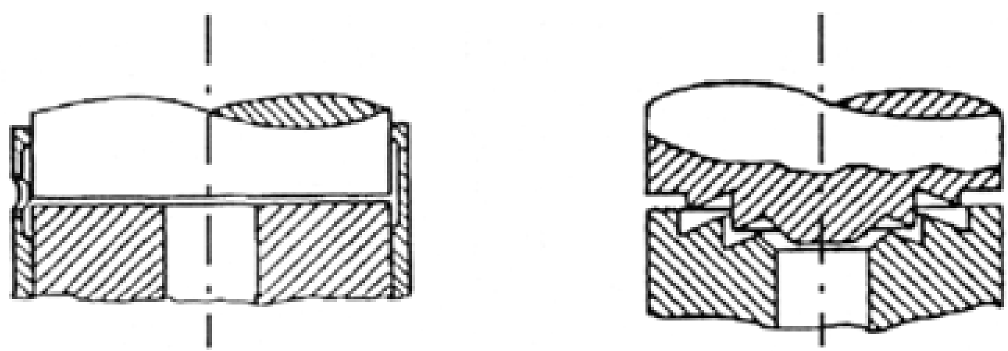


- On entering the homogenization valve, the flow speed greatly increases → pressure drops (Bernoulli) to reach the vapor pressure p_D at point A
- Since p_D is lower than the external pressure p_A → cavitation and two phase fluid flow
- Pressure signal transduction in multi-phase flows is slower than in single phase flow → equilibration with external pressure occurs late (close to exit)
- Sudden pressure jump leads to collapse of cavitational bubbles and the flow reverts to a one-phase flow
- Droplet disruption is therefore due to
  - Laminar and turbulent flow at entrance of valve (a)
  - Growth of cavitational bubbles in zone (b)
  - Collapse of bubbles in zone (c)

High Pressure Homogenizer: Efficiency Requirements

- Valve Type
  - Flat, toothed or knife edge
- Homogenization Pressure
  - Gap Size
- Counter Pressure



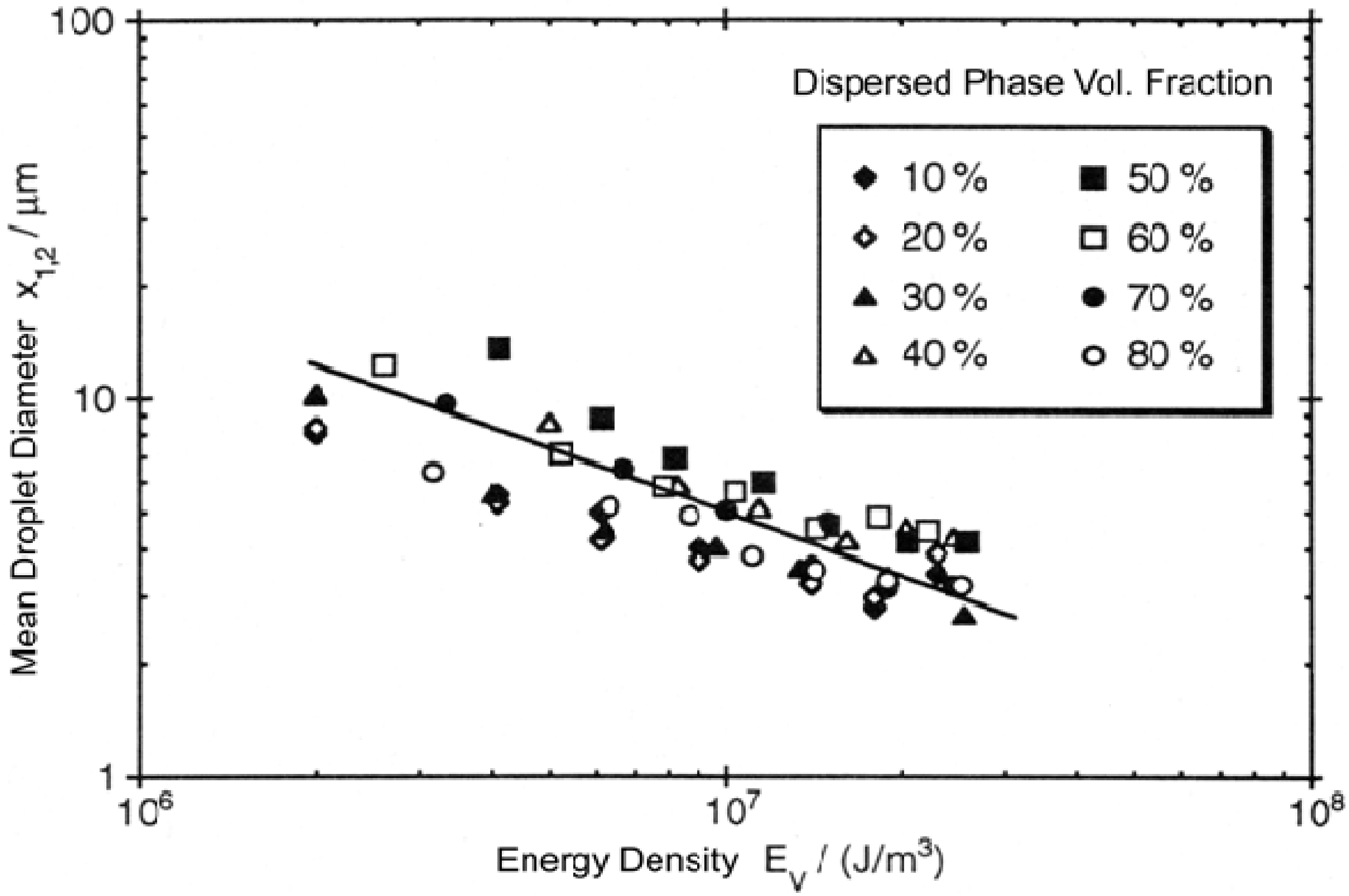


(a) (b)

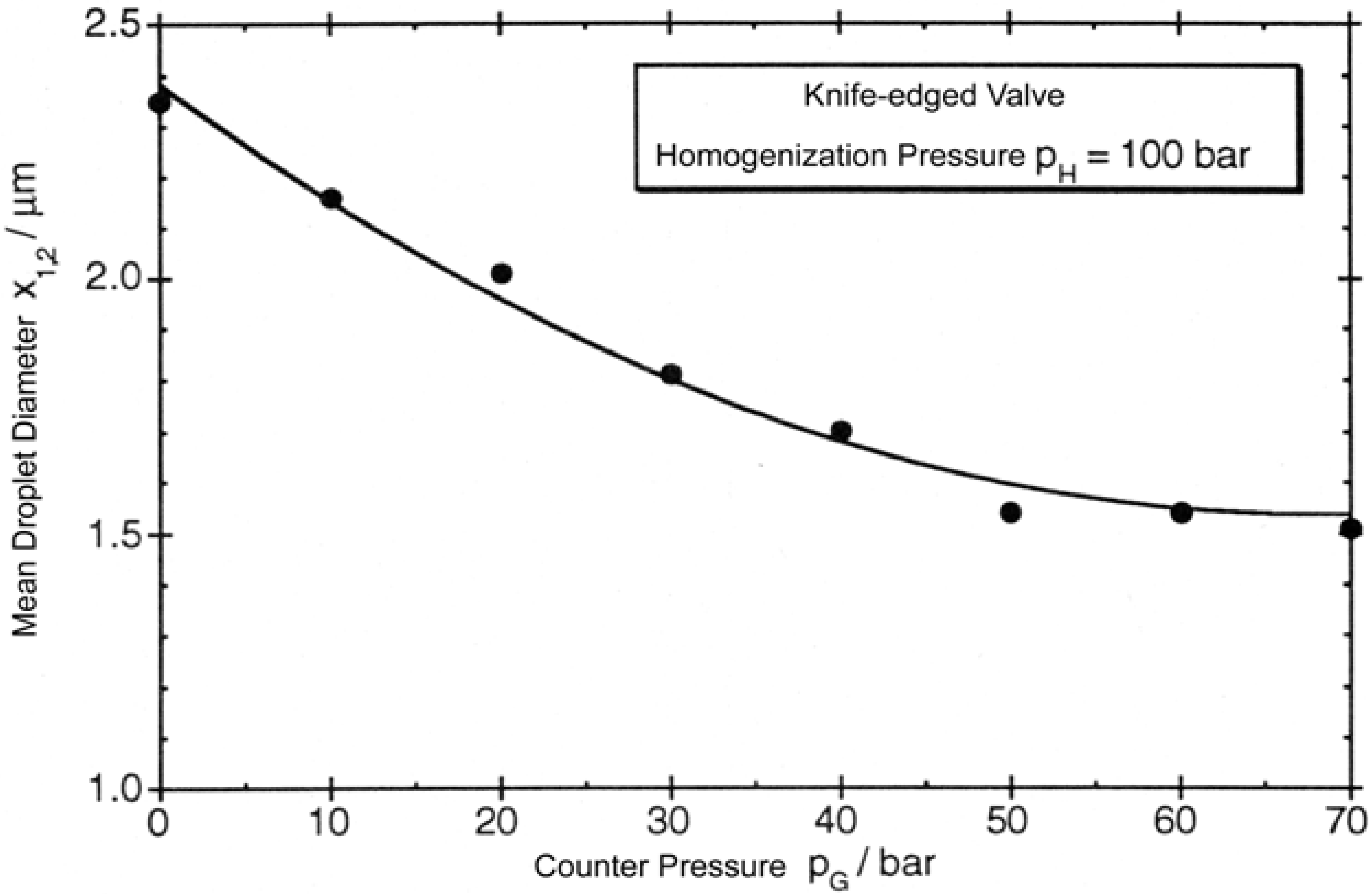
HPH Efficiency Requirements

- Homogenization valves are therefore designed with the following criteria in mind
  - Ensure sufficient time for growth of cavitational bubbles
  - “Explosive” increase of pressure towards the exit
  - Maximum height of the “Dirac-like” pressure increase
- Base process parameters for the user are homogenization pressure p_H and external (exit) pressure p_A
- The exit pressure may be increased through
  - Increased pressure in connected process equipment
  - Introduction of secondary pressure valve
  - Introduction of secondary homogenization valve

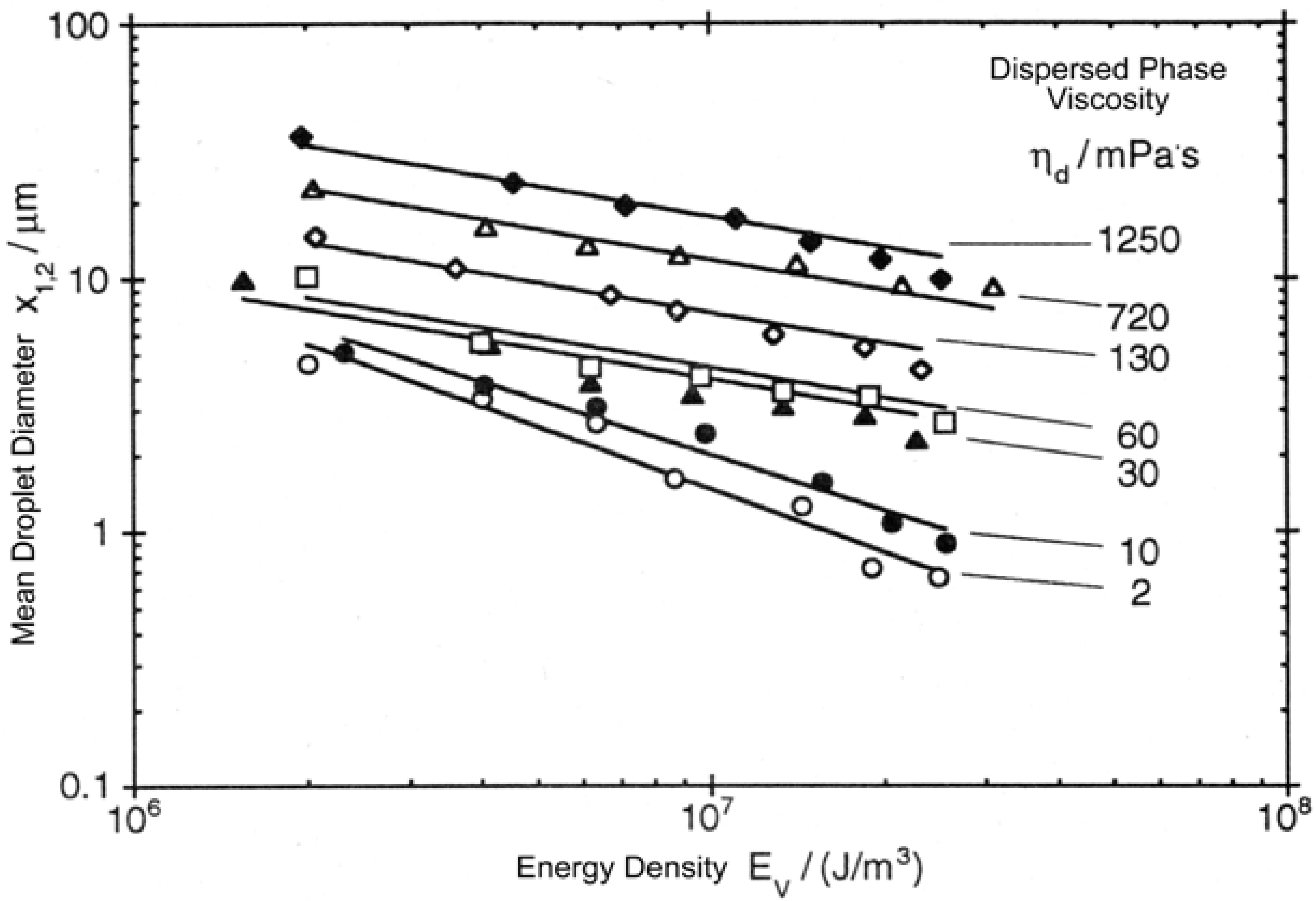
HPH: Influence of Homogenization Pressure (Energy Density)



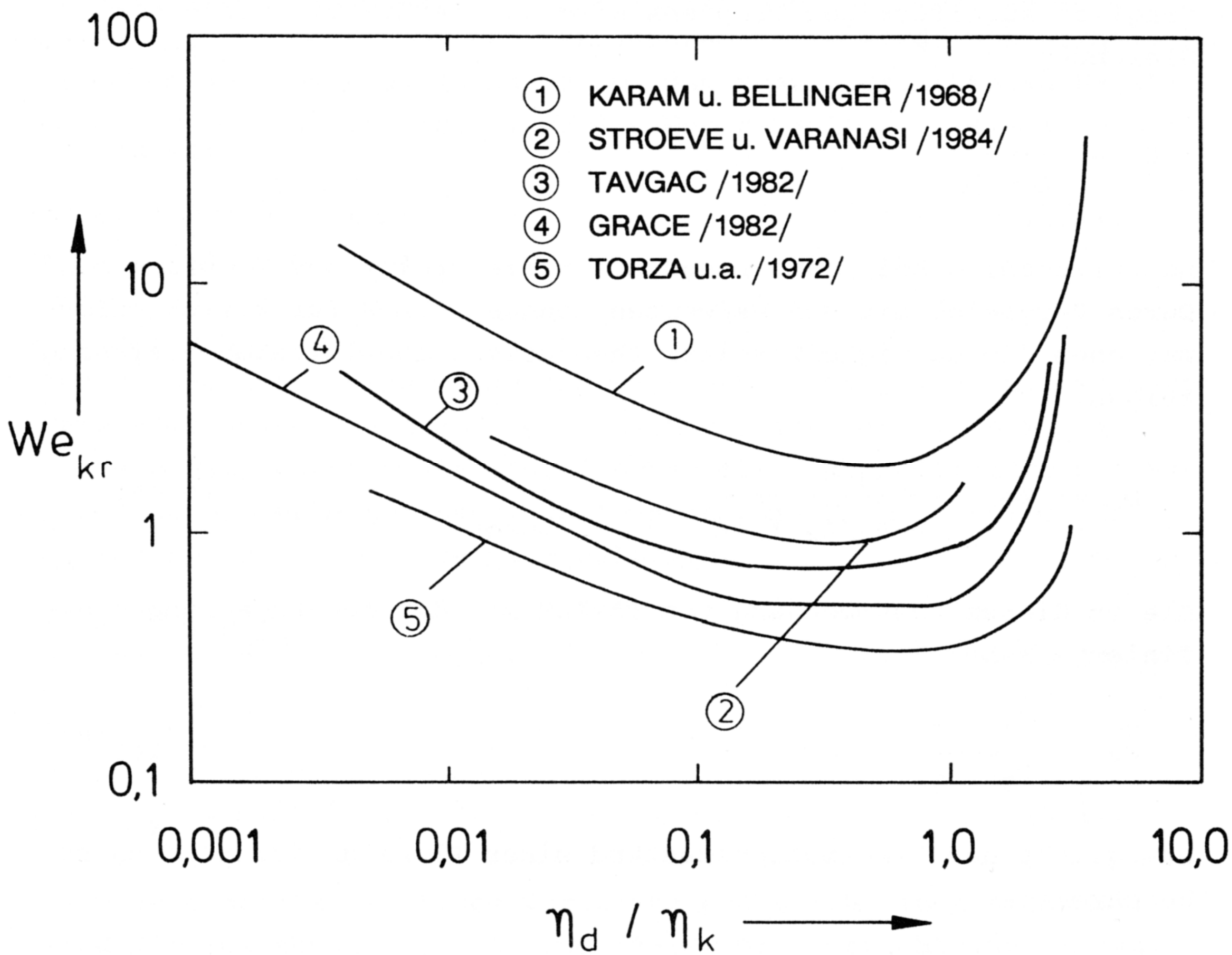
HPH: Influence of Counter Pressure



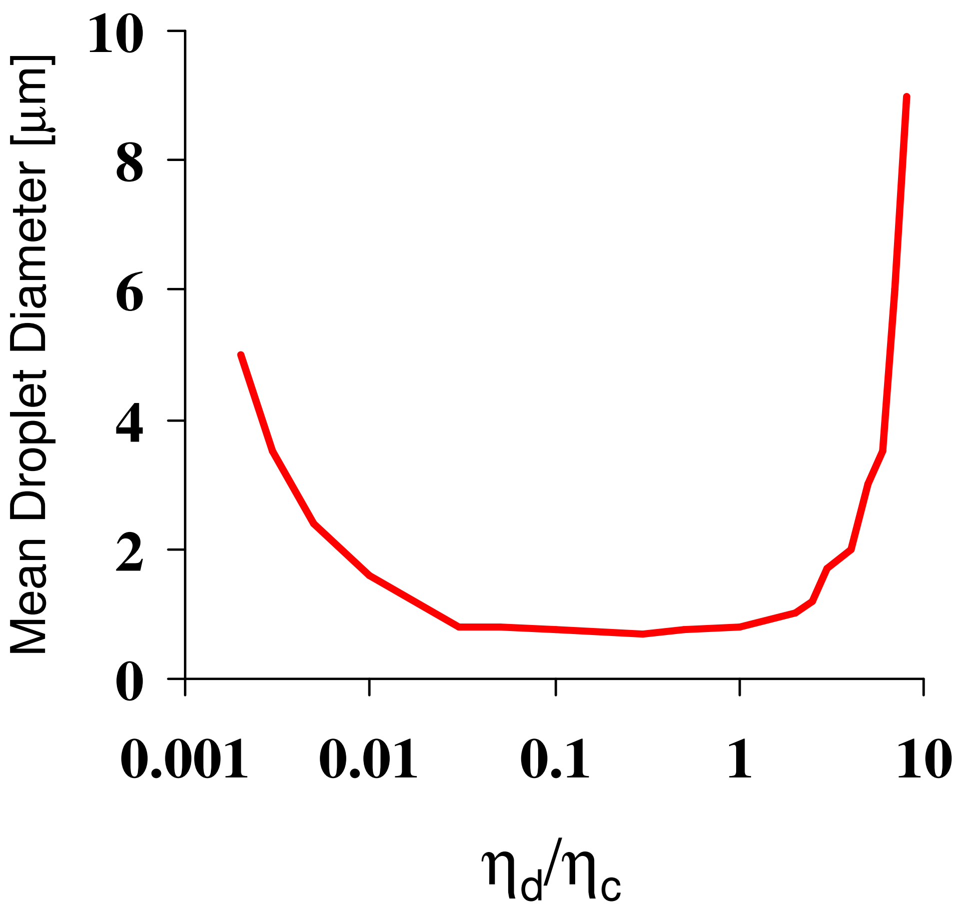
High Pressure Homogenizer: Influence of Dispersed Phase Viscosity



High Pressure Homogenizer: Influence of Viscosity Ratio



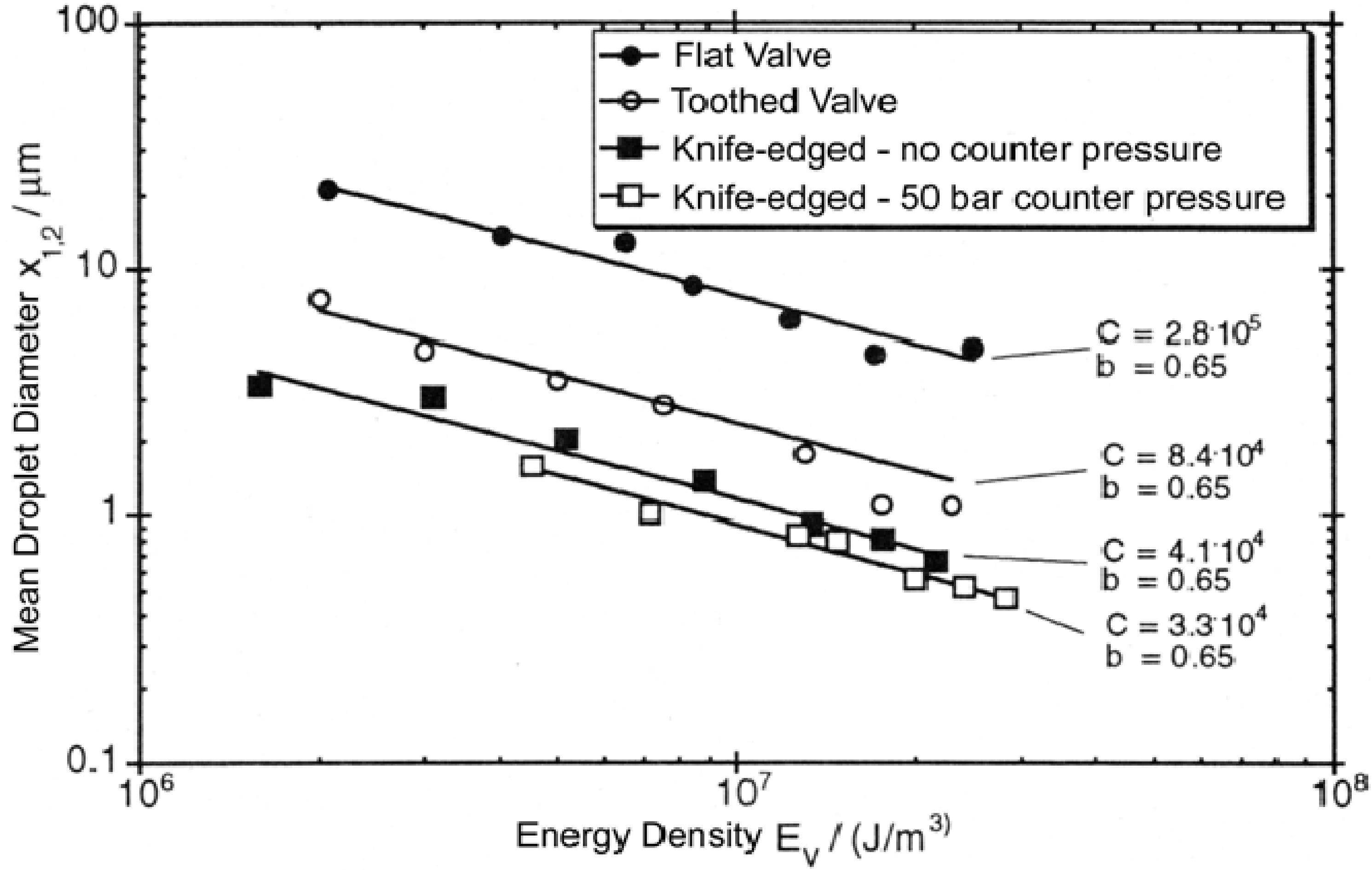
Influence of Disperse and Continuous Phase Viscosity



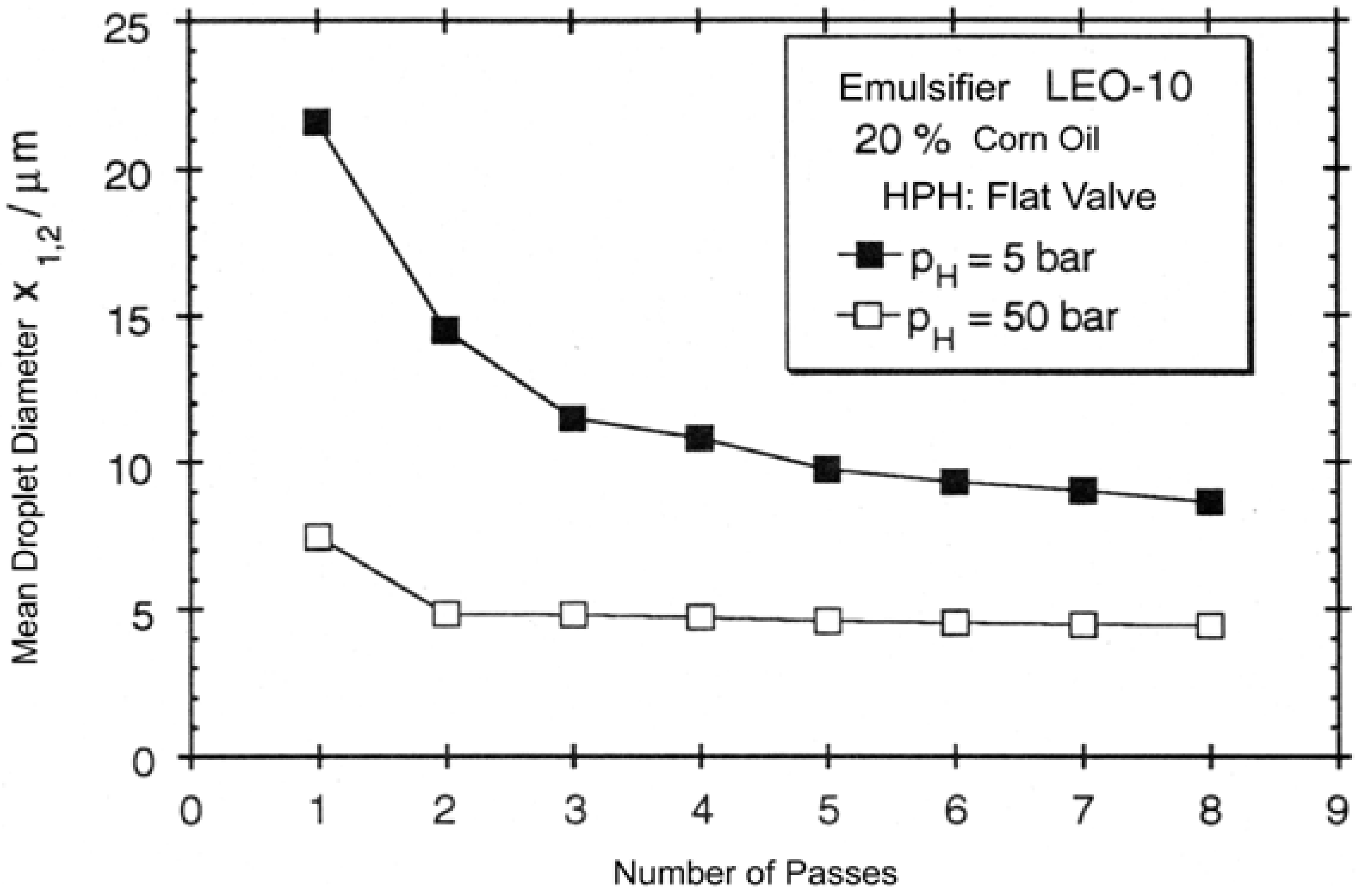
Viscosity Ratio:
Small droplets can only be formed when viscosity ratio of oil to aqueous phase is between 0.05 and 5. It is therefore important to control viscosity ratio.

Laminar Flow Conditions, O/W Emulsions, Fast Emulsifier (Schubert and Armbruster, 1992)

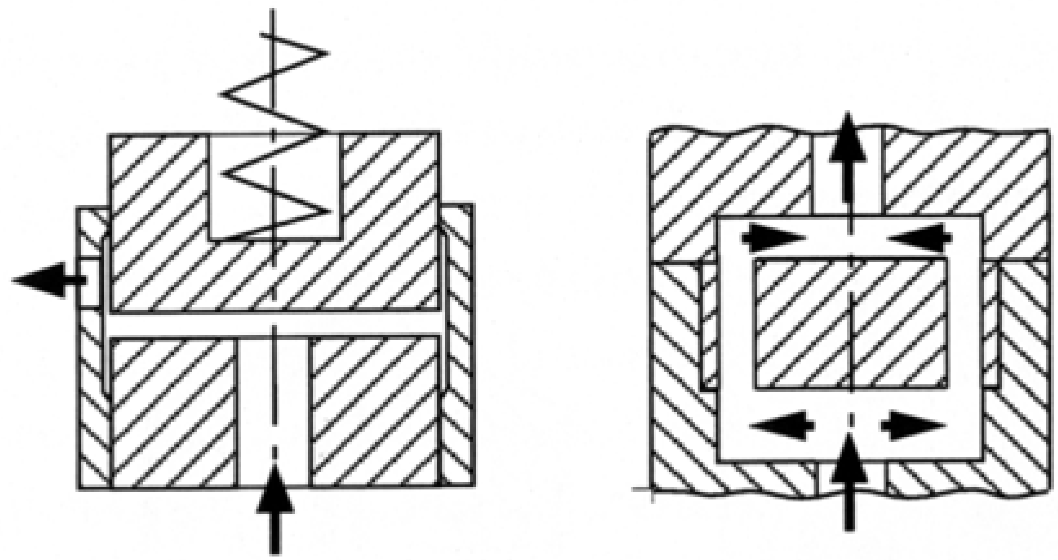
HPH:
Influence of Valve Geometry



HDH: Multistep Homogenization

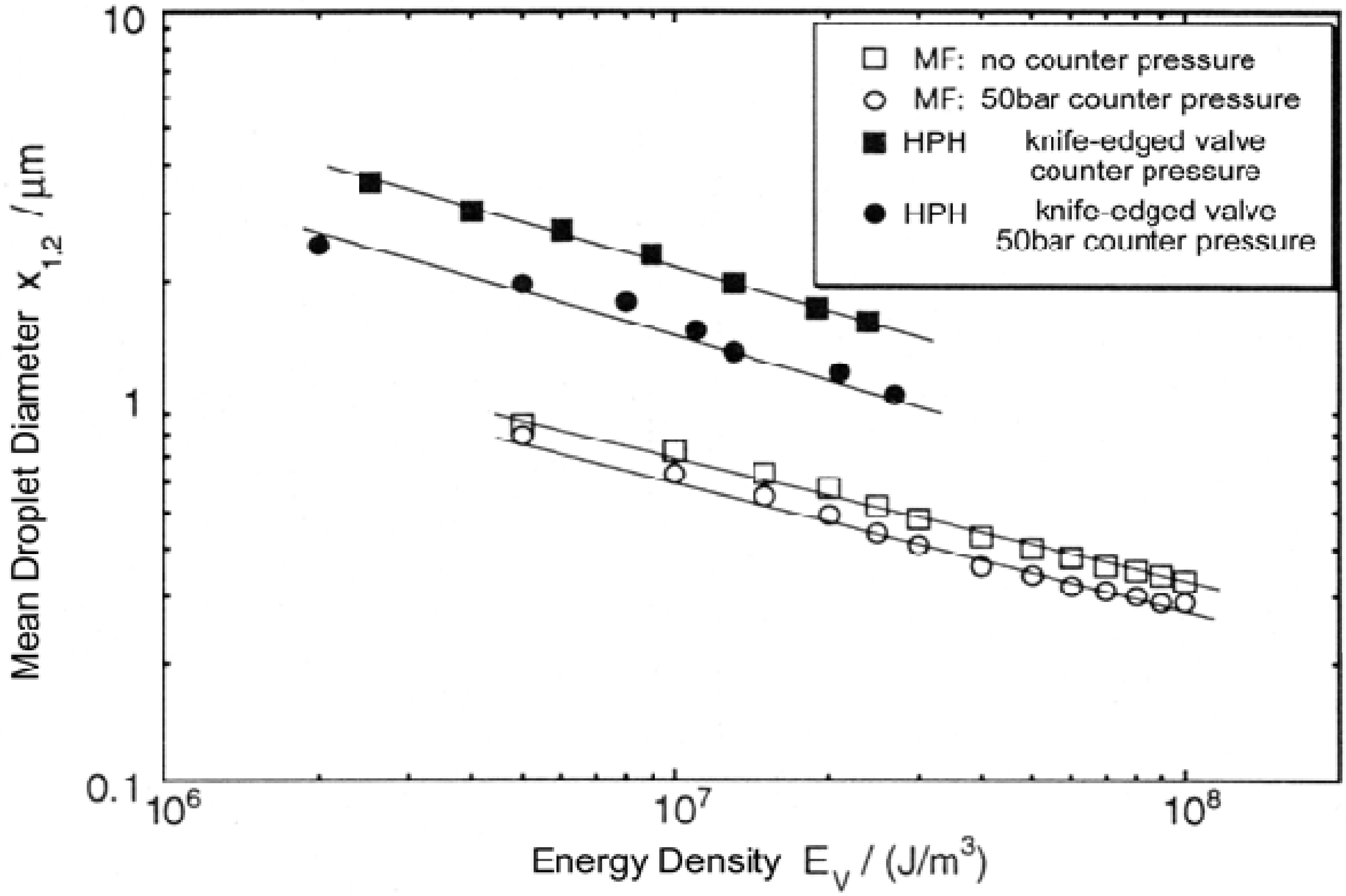


From HPH to Microfluidizer (MF)
A Small Design Change with a Large Effect



- In addition to pressure changes leading to cavitation, fluid jets also impinge in a mixing chamber
- Extremely turbulent flow situation and very high shear forces cause additional disruption of droplets
- Higher homogenization pressures possible
- NO MOVING PARTS! → low maintenance
- But: stronger possibility for blockage

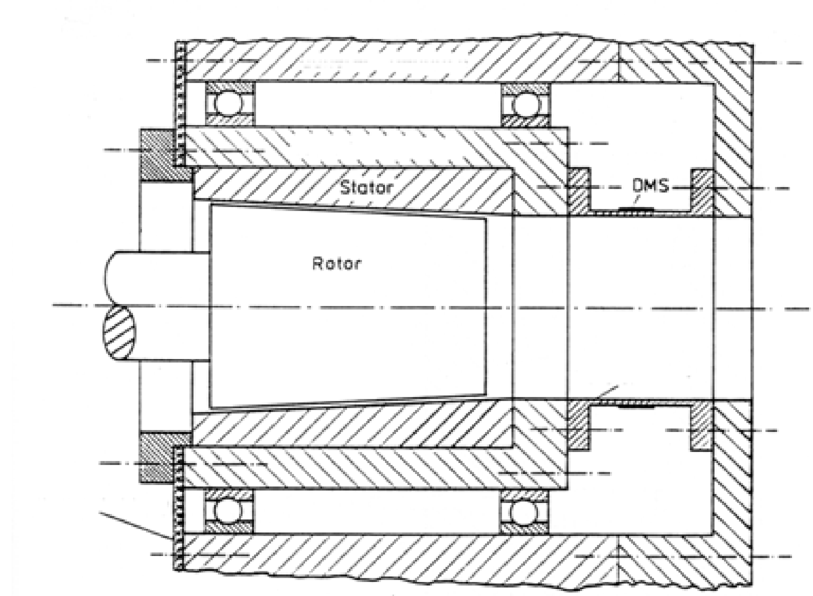
HPH and MF Comparison



III. Colloid Mills (CM)

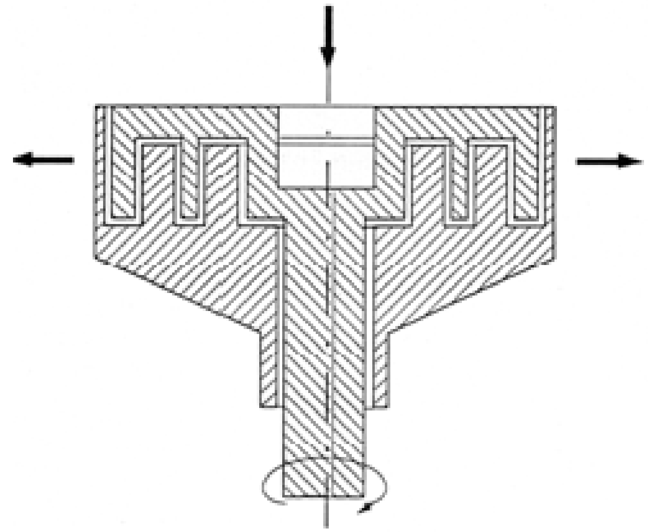
- Droplet disruption in colloid mills occurs in a flow channel between a rotor-stator assembly
- The rotor may have a large variety of surface profiles (toothed)
- Typical rotational speeds are $n=3000\ min^{-1}$

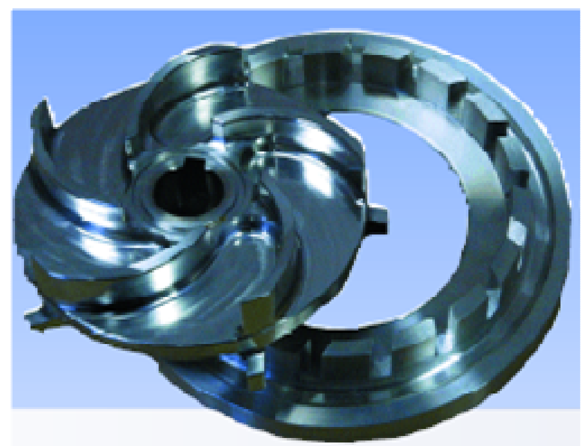
- Due to the conical design, the emulsions is transported without external pressure application
- The size of the gap alters residence time in the channel and exerted forces



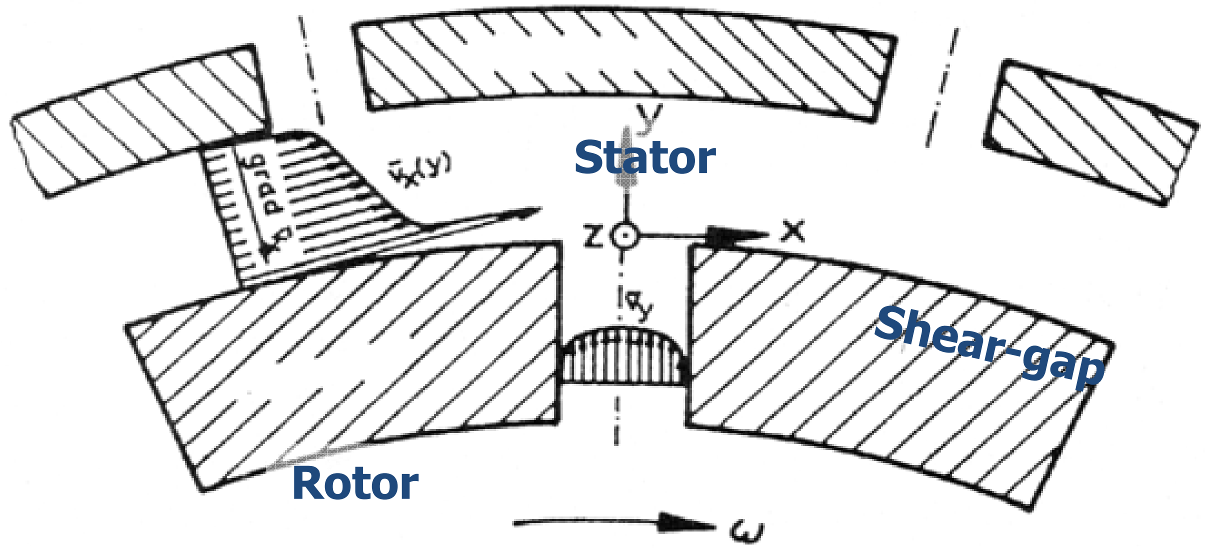
IV: High Shear Disperser (HSD) – (Ultra) Turrax

- HSD or Ultra-Turrax Systems are rotor-stator systems similar to colloid mills
- They are composed of coaxial intermeshing rings with radial openings
- The fluid enters in the center of the systems and is accelerated by the rotor
- As it passes through the system, the fluid is accelerated and decelerated multiple times which results in high tangential forces
- Rotational speeds may be as high as $n=20000\ \text{min}^{-1}$



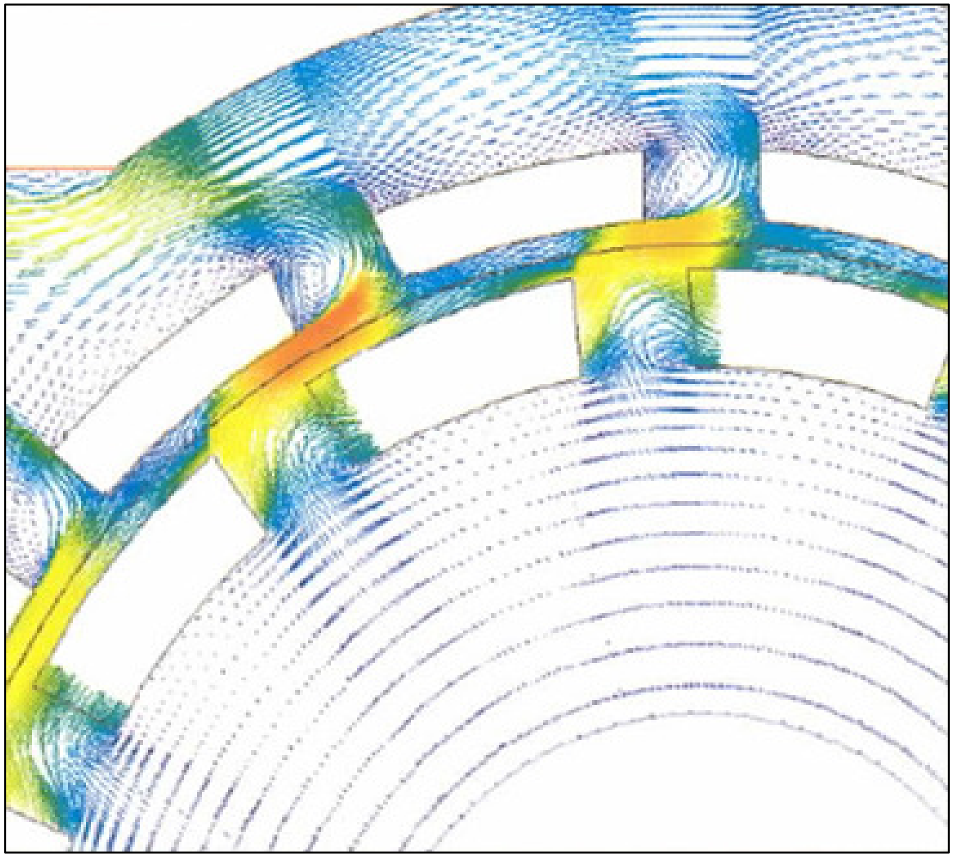


High Shear Disperser (HSD): Principle of Droplet Disruption

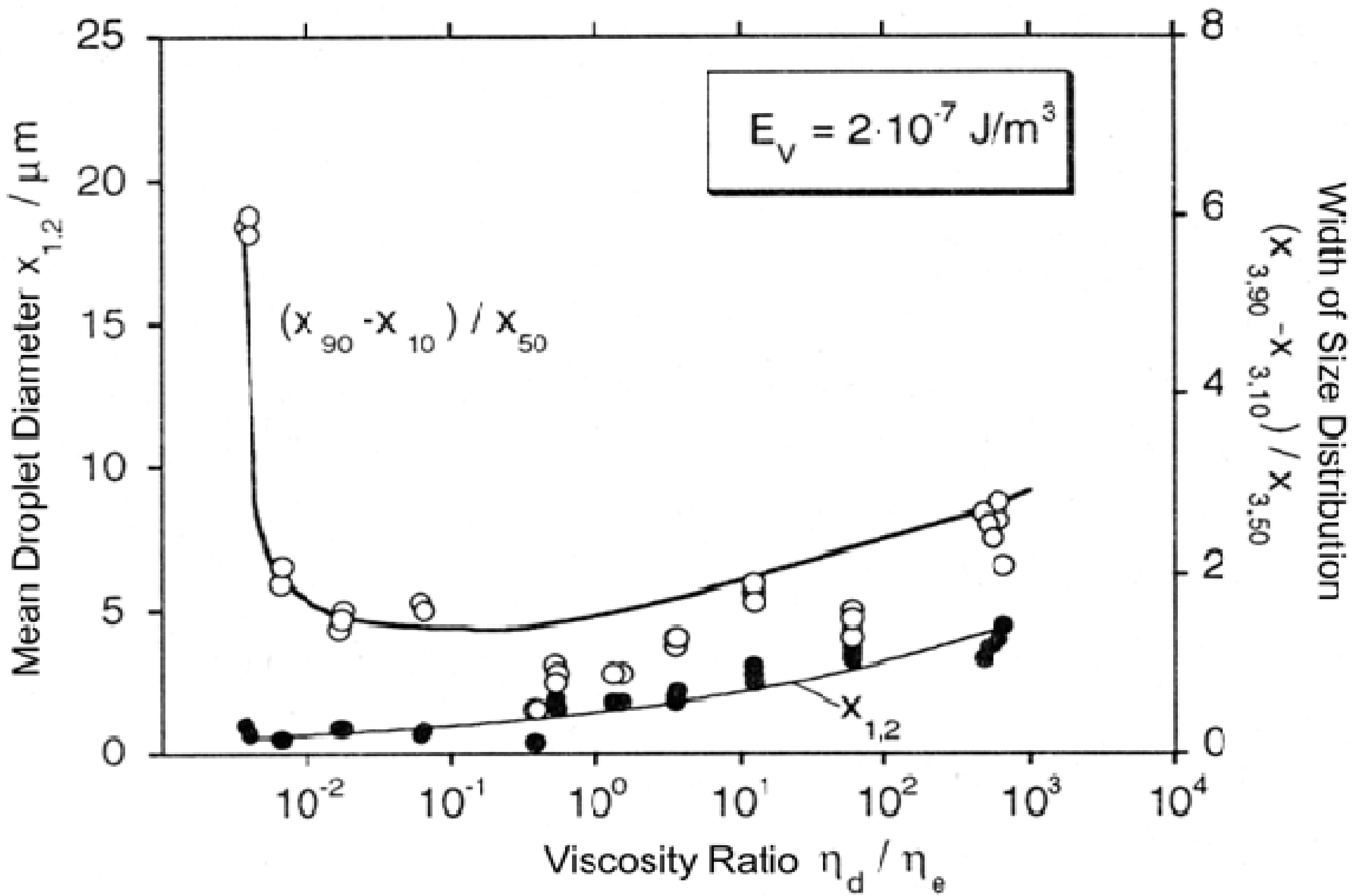


- The pre-emulsion is transported through the radial openings of the rotor systems and mixes with the liquid in the gap between the rotor and the stator
- High shear forces and turbulent flow situations arise that lead to a disruption of droplets

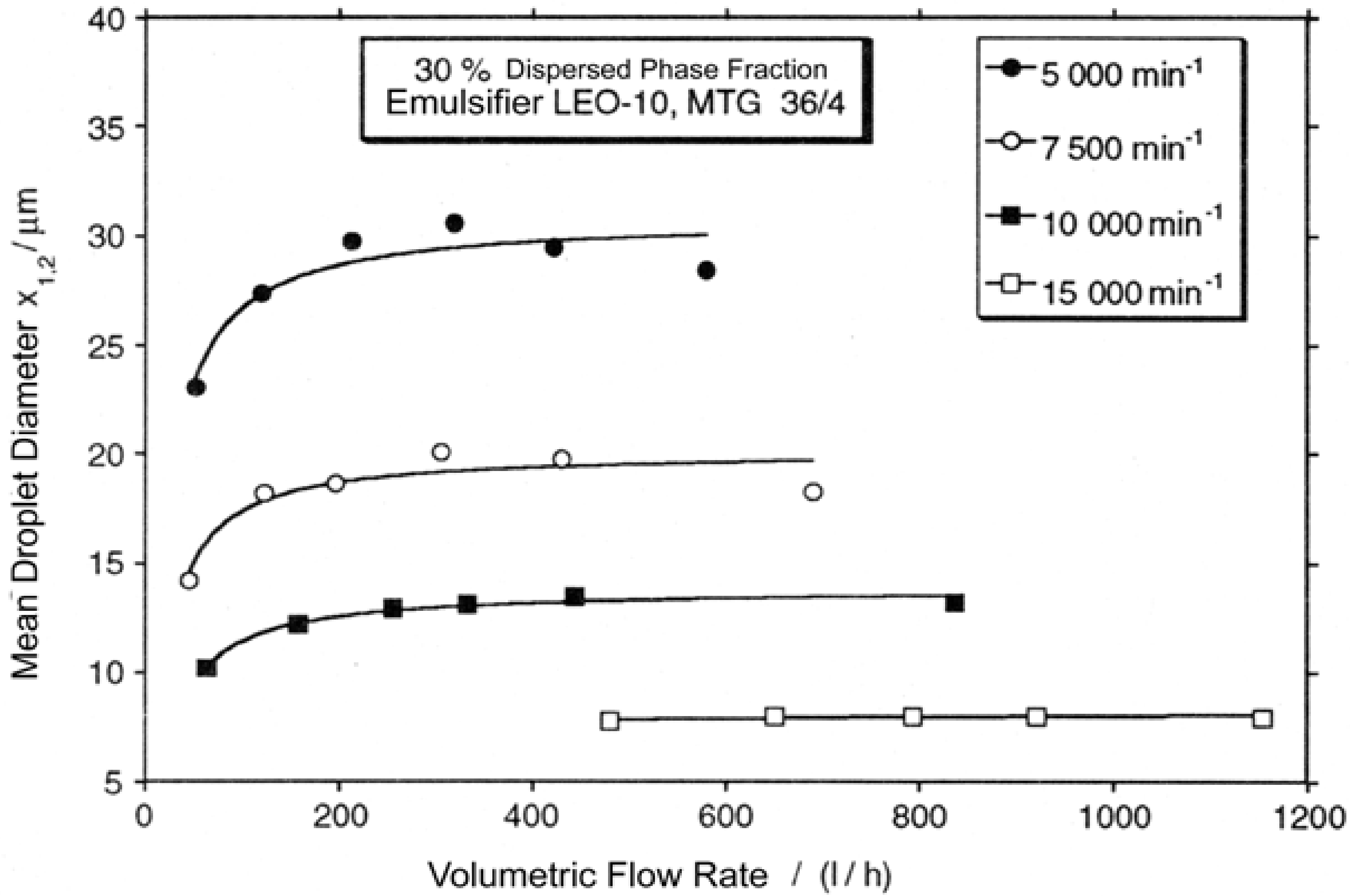
HSD:
Complexity of Flow Profiles



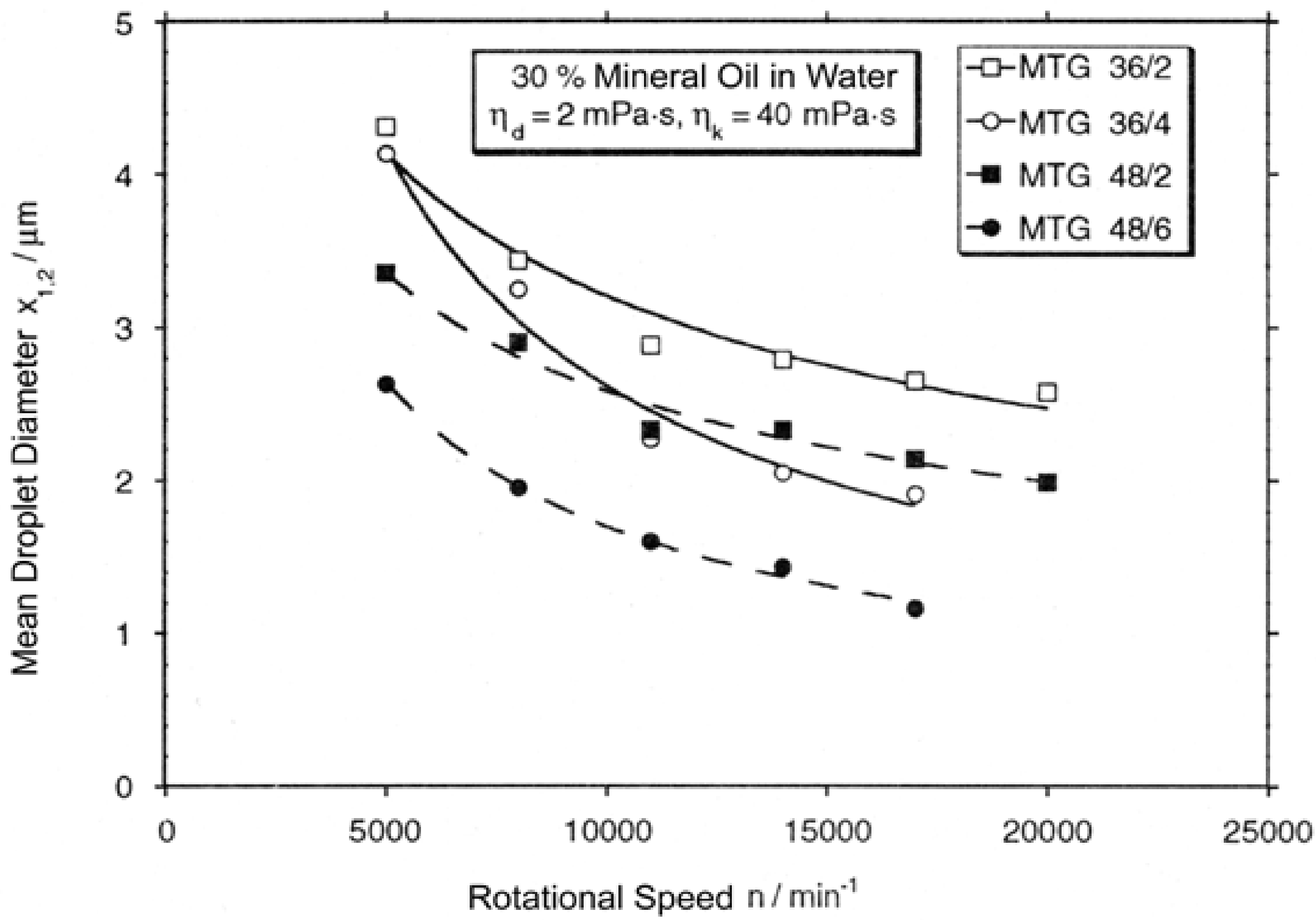
HSD: Influence of Viscosity Ratio



HSD:
Influence of Residence Time

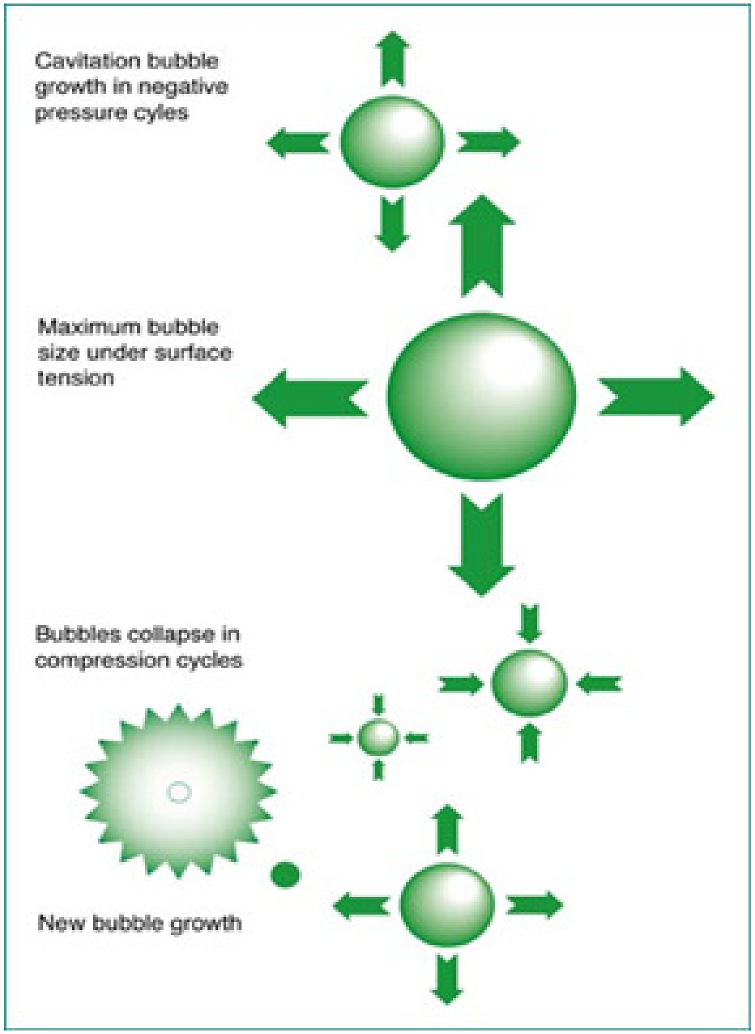


HSD: Influence of Geometry of Rotor-Stator System

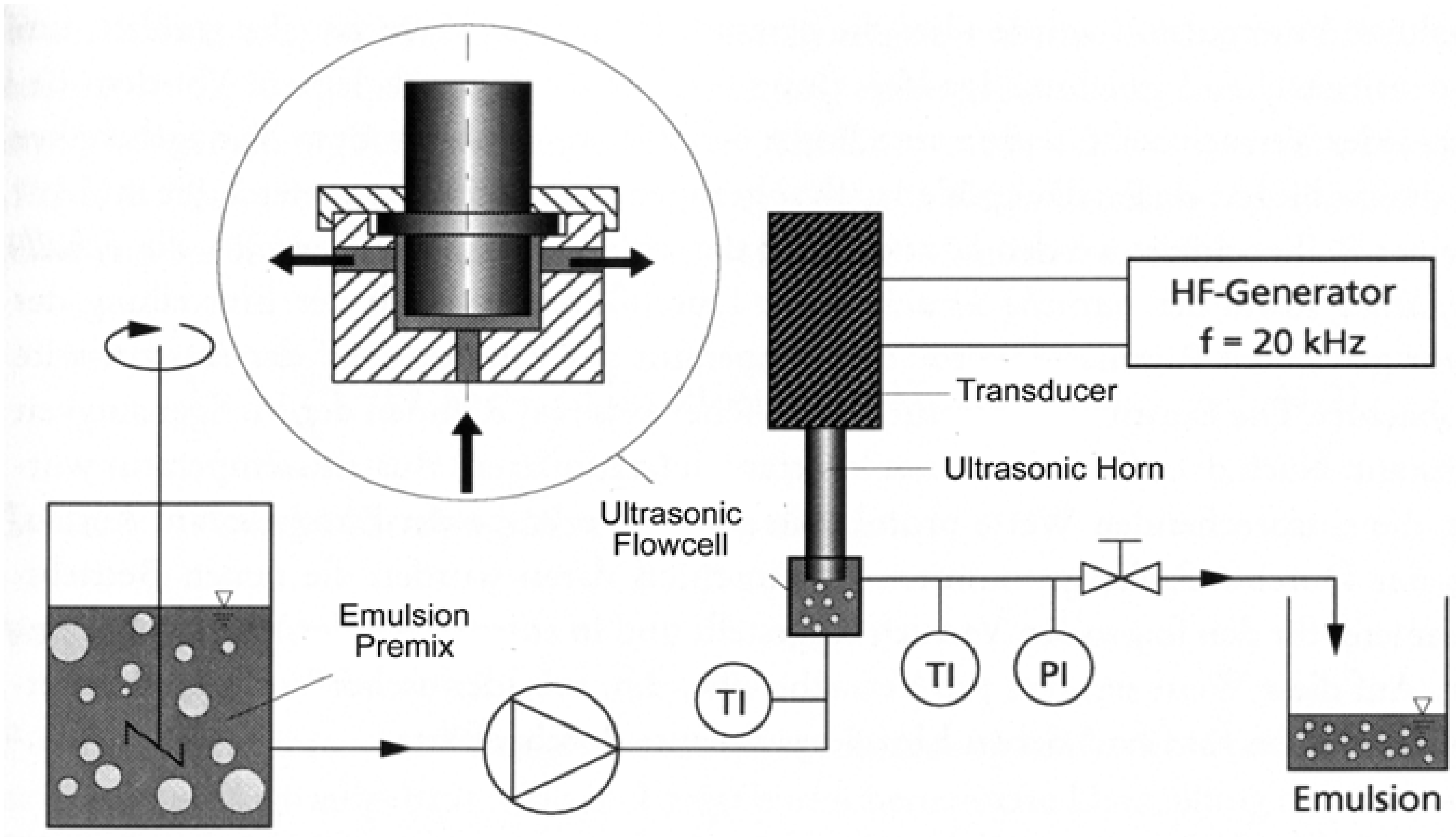


V. Ultrasonic Homogenizer (UH)

- Piezo-electric or magneto-restrictive transducers generate sound waves with frequencies > 20 kHz
- Associated pressure gradients cause deformation of droplets
- Cavitation caused by drop of local pressure below the vapor pressure of the solvent generates turbulent flow and high shear
- Can be run in batch or continuous using a flow cell



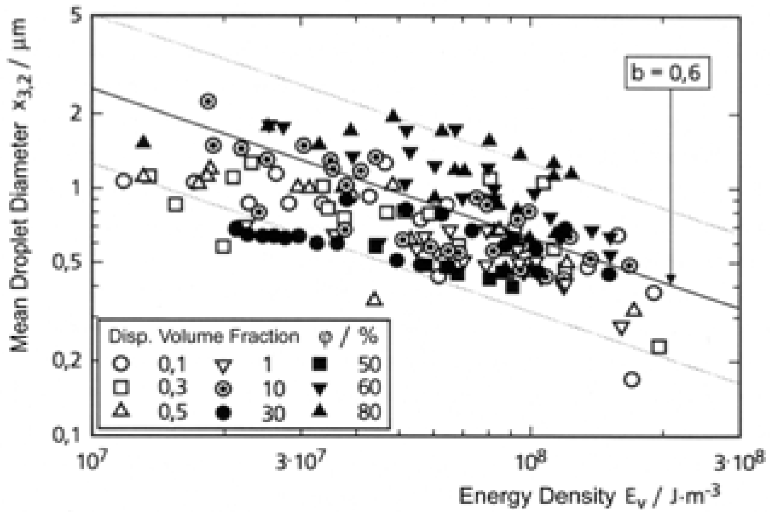
Typical Design of a Continuous Ultrasonic Homogenizer

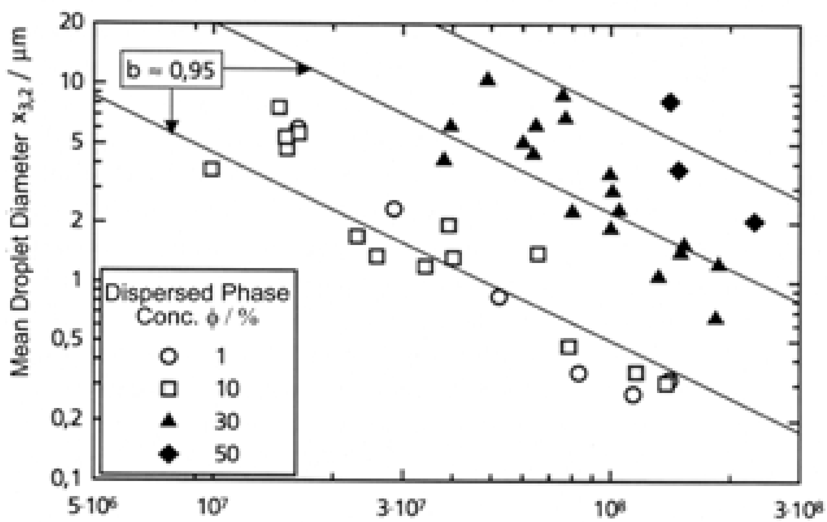


Ultrasonic Homogenizer : Process Parameters

- **Frequency**
  - Size of cavitational bubbles decreases with increasing frequency
  - Wave intensity
  - A minimum pressure drop is required to initiate cavitation → below critical wave intensities, almost no droplet disruption
  - Above the critical pressure, direct relationship between applied intensity and achievable particle size
- **Sonication time**
  - Disruption and stabilization are kinetic events, thus, a minimum sonication time is required to achieve droplet disruption
- **Hydrostatic pressure**
  - Initially, improved emulsification (up to 5 bar), above 5 bar, decreased efficiency
- **Dissolved gas concentration**
  - Increased number of cavitational events but reduced intensity of cavitational collapse due to dampening
- **Viscosity & Temperature**
  - Influences droplet disruption

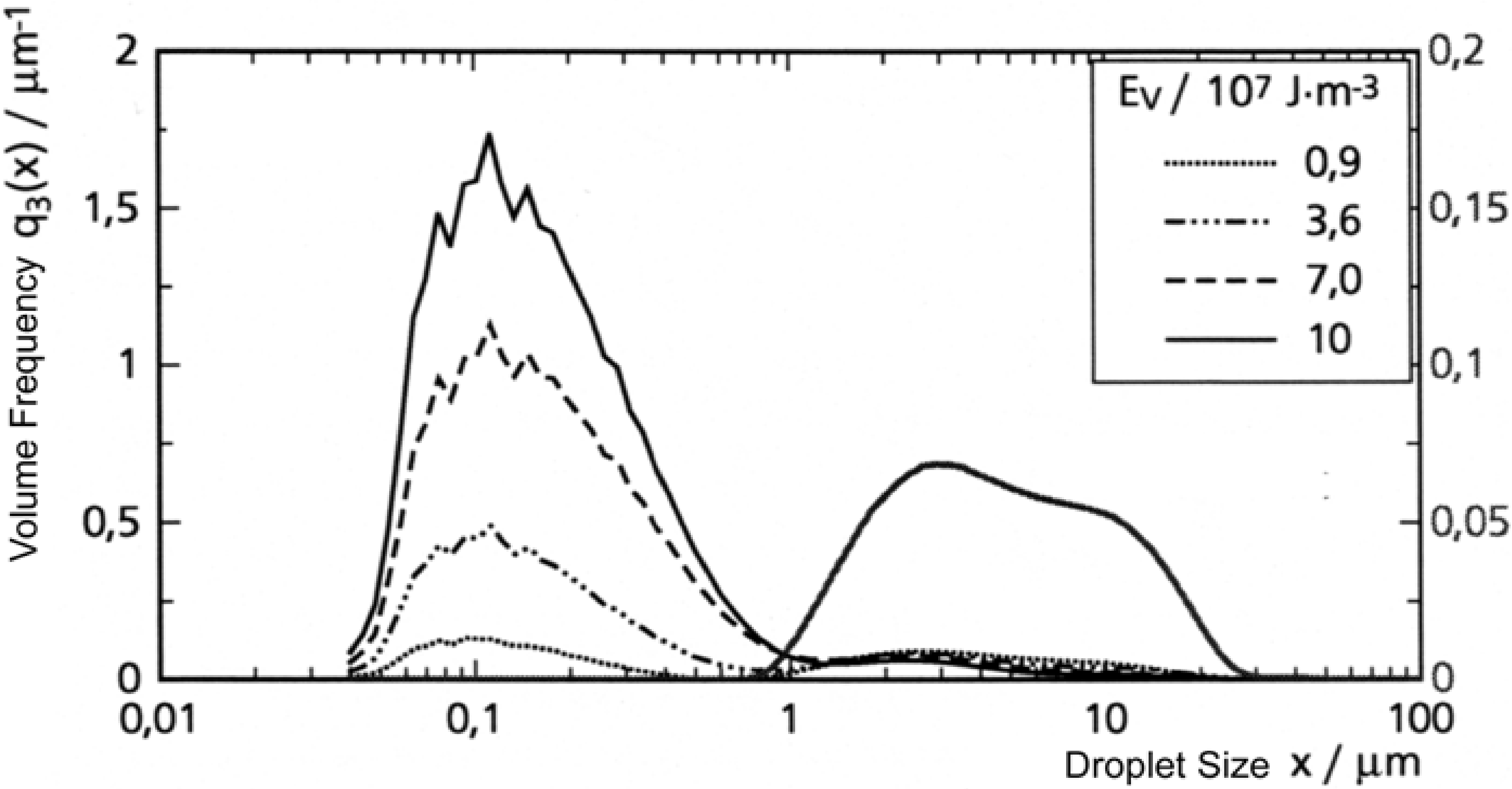
Ultrasonic Homogenizer: Emulsifier Properties Play a Key Role



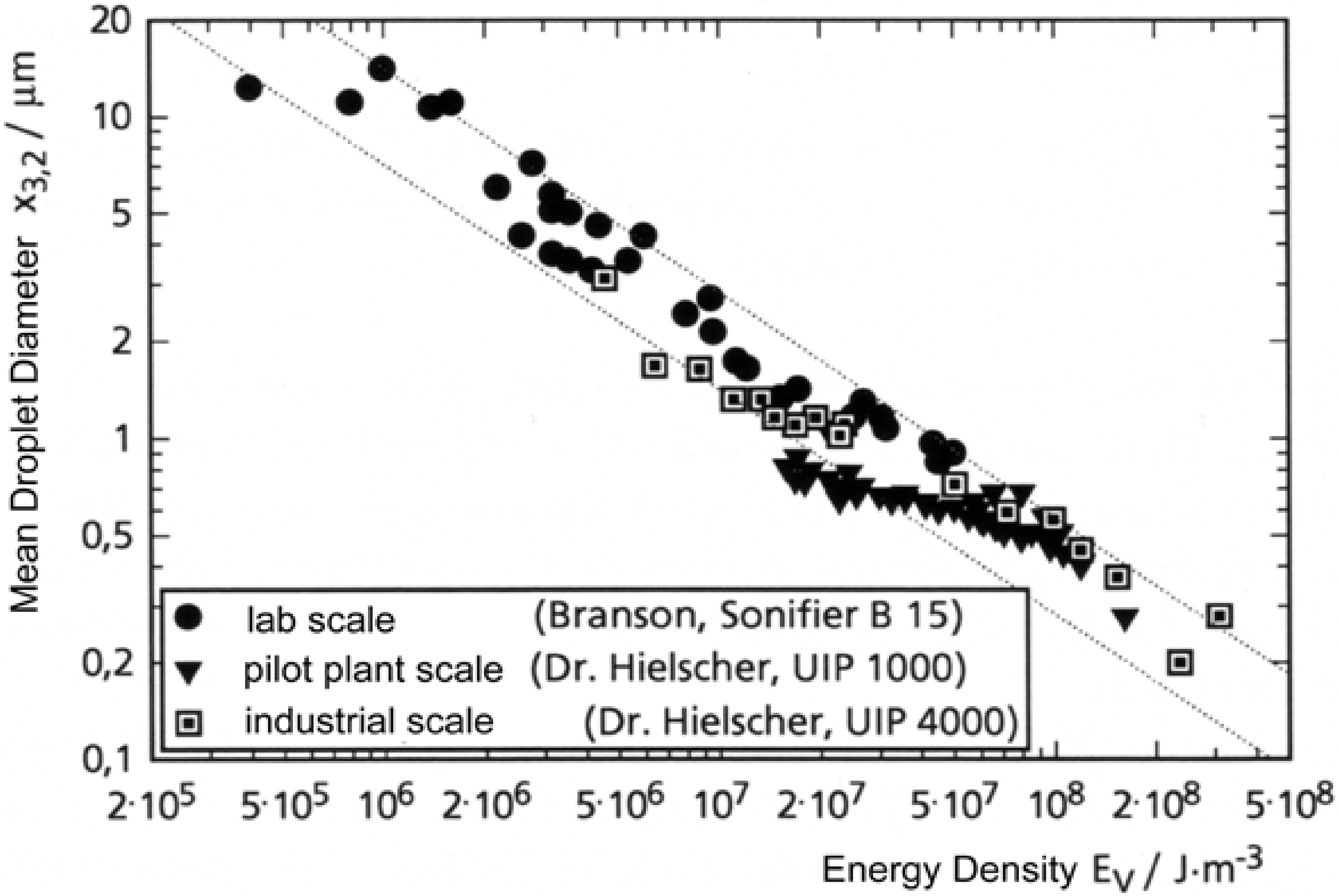


- The relationship between energy density, volume fraction and achievable mean droplet diameter depends strongly on the emulsifier.
- For slow emulsifiers (bottom), achievable particle sizes increase with increasing volume fraction
- For fast emulsifiers (top), smaller emulsion can be obtained even at high dispersed phase concentrations

Ultrasonic Homogenizer: Peculiarity in Size Distribution Development

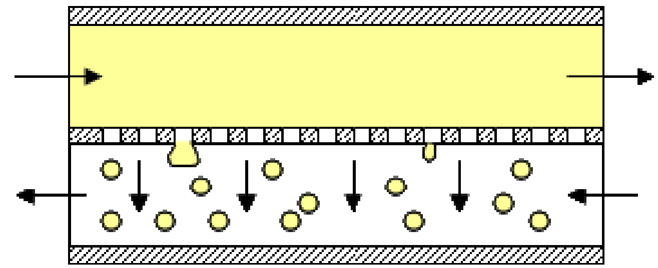


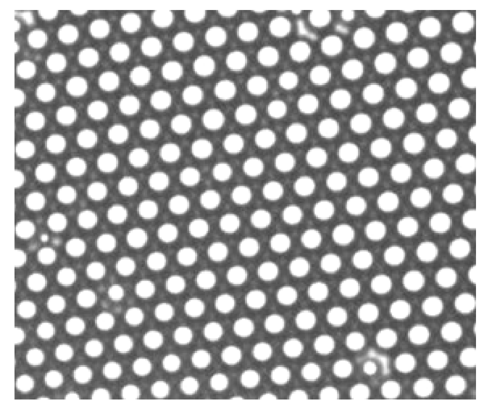
Ultrasonic Homogenizer: Scale-up of Homogenizers



VI. Membrane Homogenizers (MH)

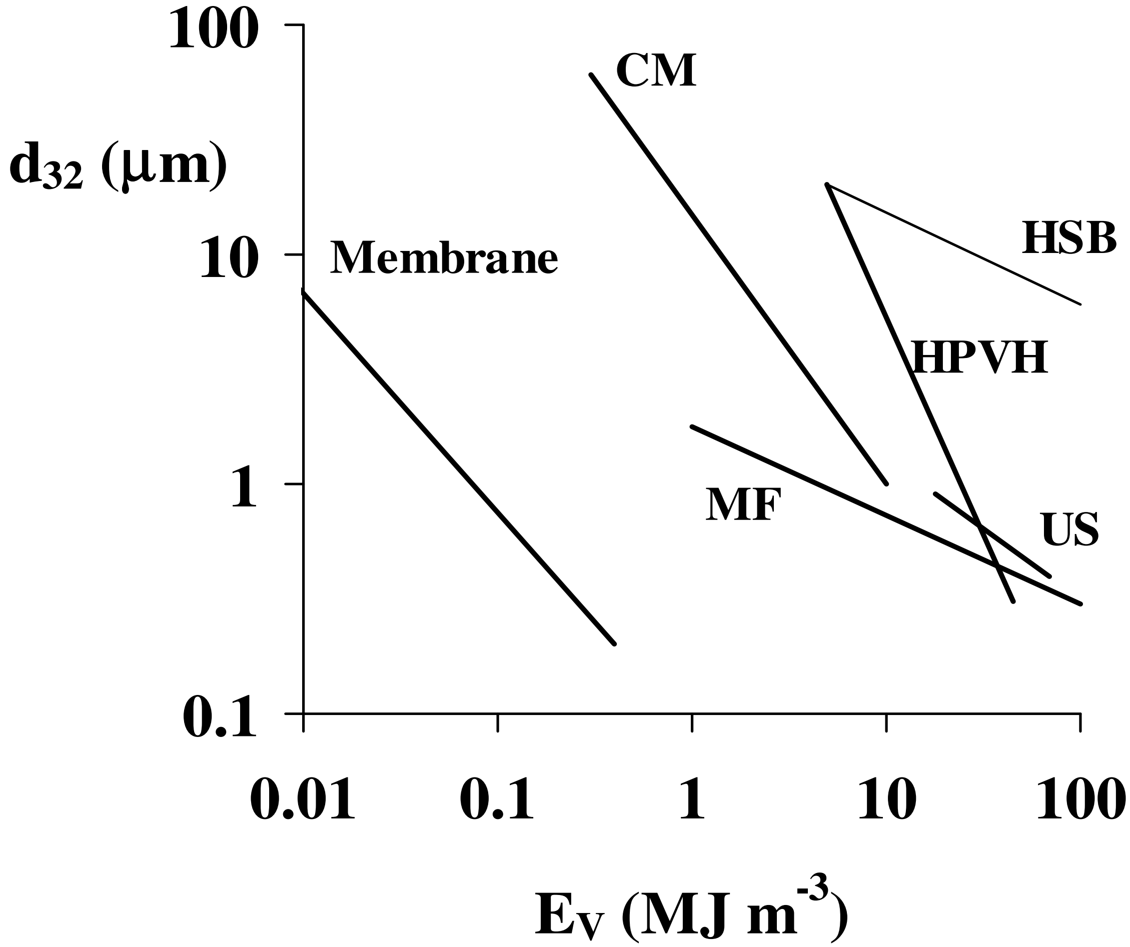
- Dispersed phase or emulsion pushed through microporous membrane into continuous phase
- High energy efficiency
- Narrow particle size distribution possible
- Droplet size typically 2-3 $\times$ pore size
- For high dispersed phase concentrations, recirculation is needed → time consuming
- Process parameters include:
  - Membrane pore size and material
  - Volume flow rates
  - Applied trans-membrane pressure
  - Others: temperature, viscosity, etc.





O/W Produced by Membrane

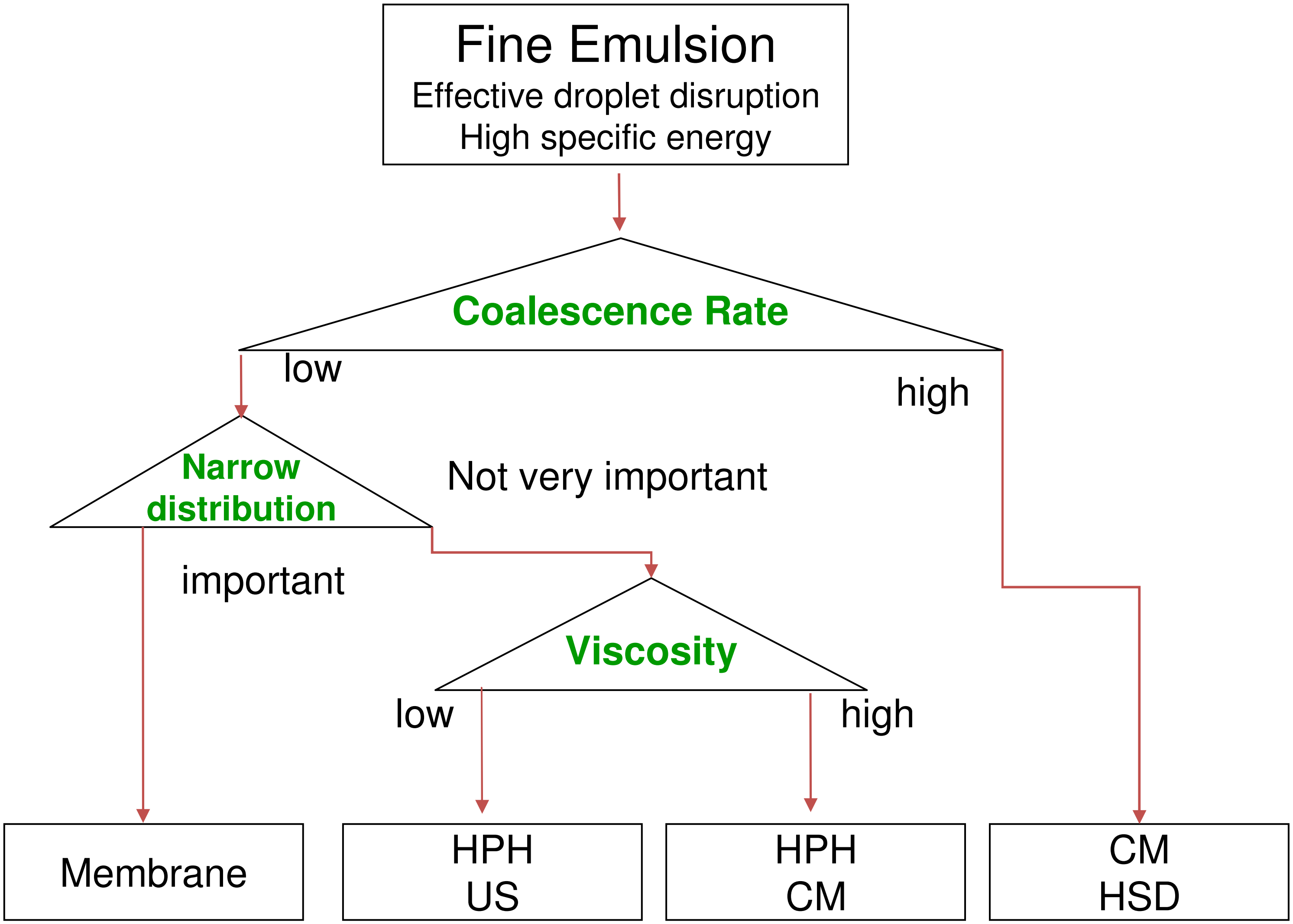
Direct Comparison of Homogenizers



Selecting a Homogenizer

- Define desired product characteristics
  - Particle size distribution, viscosity
- Define desired production conditions
  - Batch/Continuous, throughput, hygiene, temperature
- Identify, Test & Compare Homogenizers
  - High speed blender, high pressure valve, colloid mill, ultrasonic, membrane etc.
- Optimize Homogenization Conditions
  - Pressure, flow rate, rotation speed, time, temperature, emulsifier type, emulsifier concentration

Which Homogenizer to Buy?



Recap:
Factors Affecting Droplet Size

- **Homogenizer**
  - Machine Parameters (Pressure, Rotation Speed, Time)
  - Energy Density
- **Component Phases**
  - Interfacial Tension
  - Viscosity
- **Emulsifier**
  - Adsorption Kinetics
  - Interfacial Tension Reduction
  - Stabilization of Droplets Against Aggregation