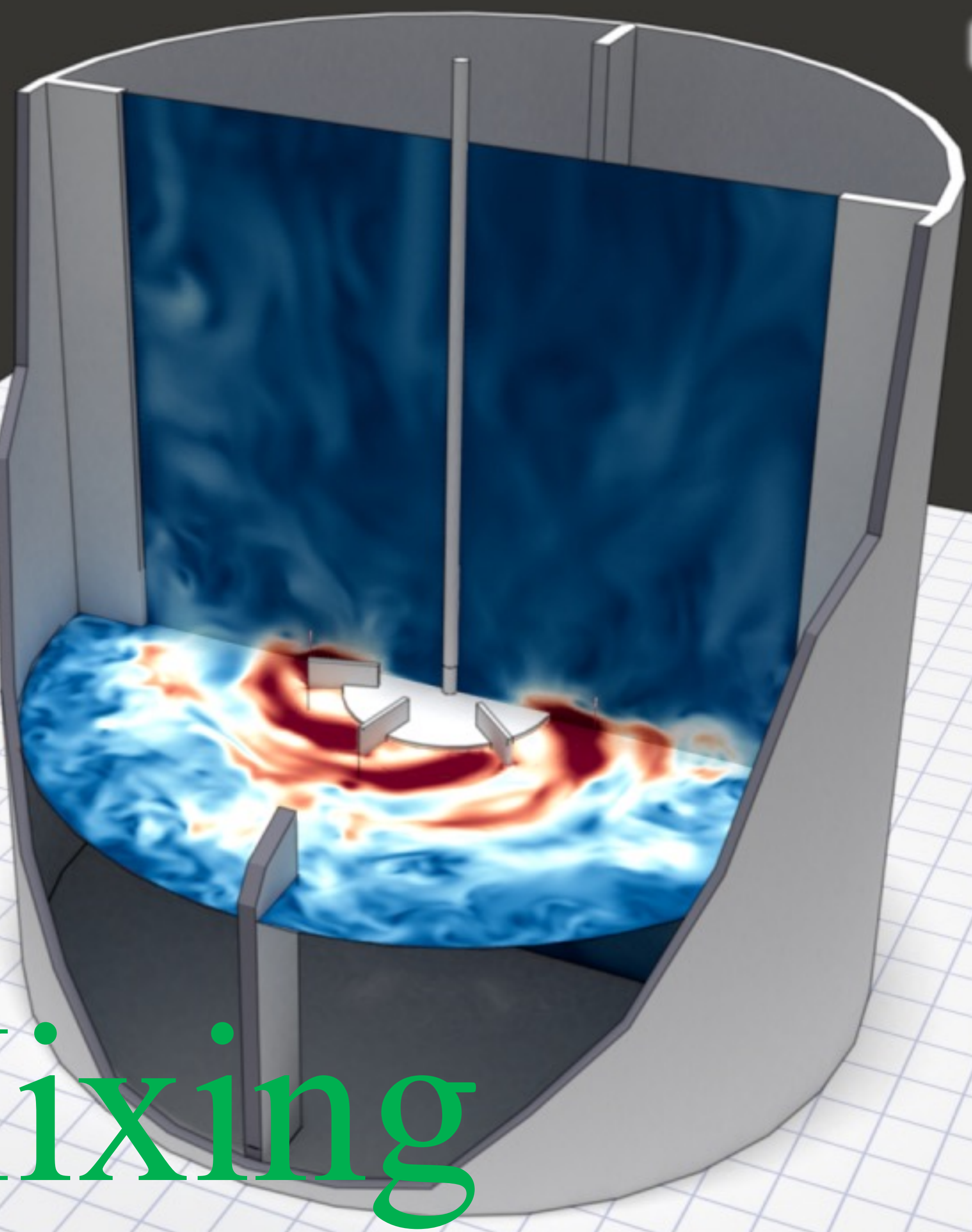
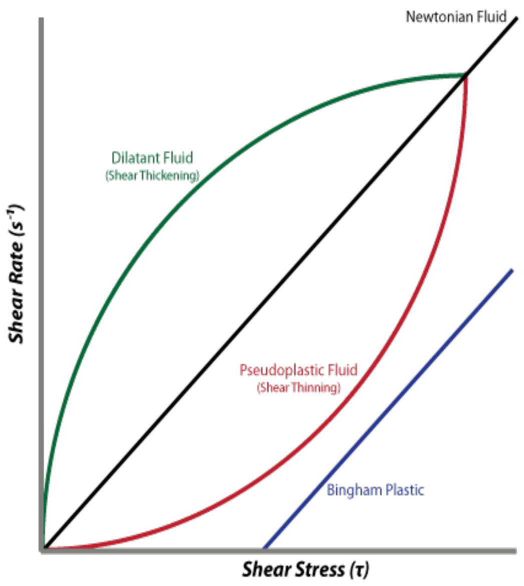
Rheologic



Fluid Mixing

Flow Characteristics

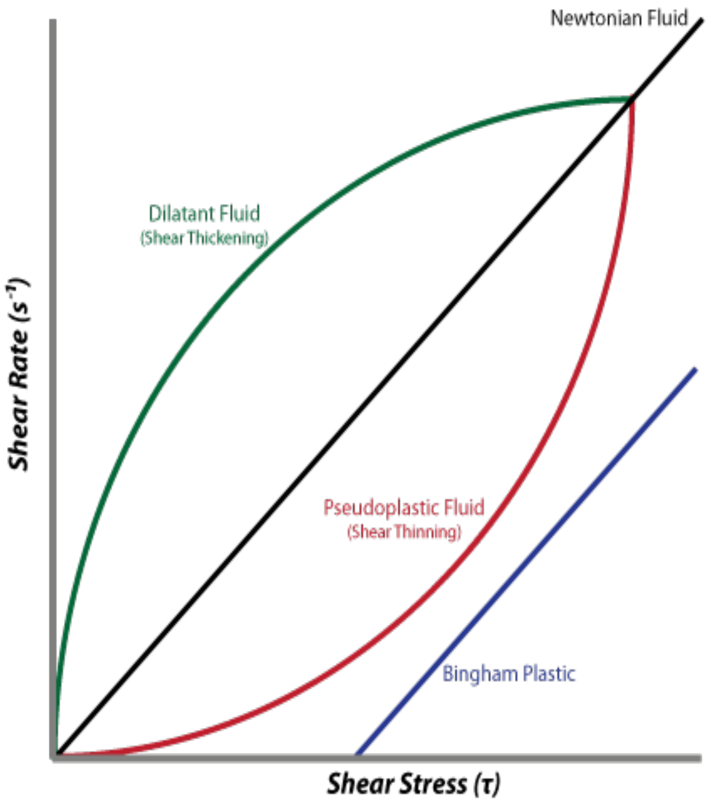
- Fluids may be generally classified as **Newtonian and non-Newtonian**, depending on the relationship between their shear rates and the applied stress.
- The rate of shear may be defined as the derivative of velocity with respect to distance measured normal to the direction of flow.
- The **viscosity** is the ratio of shear stress to the shear rate.
- **Forces of shear** are generated by interactions between moving fluids and the surfaces over which they flow during mixing.



Flow Characteristics

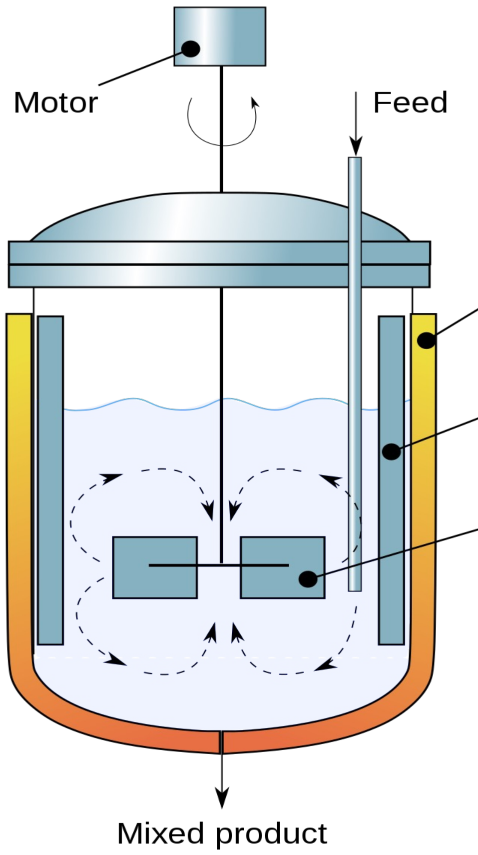
- **Newtonian Fluid**:
- The rate of shear is **proportional to** the applied stress, and such fluids have a dynamic viscosity that is **independent** of the flow rate.
- Like **water** where constant viscosity; ➔ the rate of shear increases with shear stress
- **Non-Newtonian fluid**:
- Apparent dynamic viscosity is a function of the shear stress. Such as

1. **Plastic flow**: a fluid **after a certain shear stress limit**
2. **Pseudoplastic flow**: (shear thinning behavior)
3. **Dilatant flow** (shear thickening behavior)



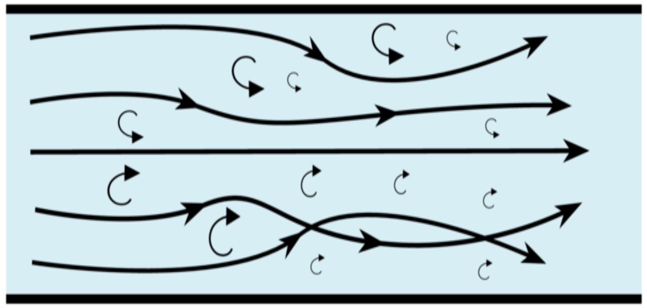
Liquid Mixing Mechanisms

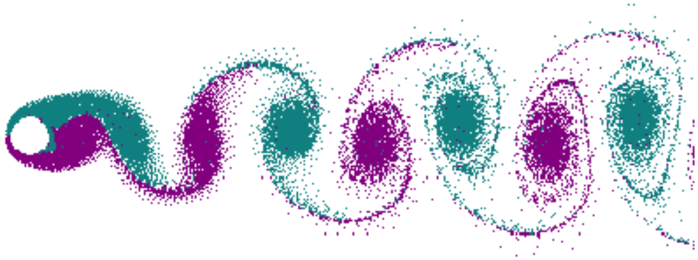
- **Mixing Mechanisms:** can be divided into four mechanisms and usually **more than one** of them occurs in the mixing of liquid:
- **First: Bulk transport**
  - Movement of a **large portion** of liquid to be mixed from one location to another.
  - However, this mechanism leaves a part of the liquid within the moving material unmixed.
  - ➔ A simple circulation of material **does not necessarily** result in efficient mixing.
- This mechanism is usually accomplished using paddles, revolving blades, and other devices.



Liquid Mixing Mechanisms

- **Second: Turbulent mixing**
- This resulted directly from the **turbulent fluid flow** which is characterized by the random fluctuation of fluid velocities at any given point within the system.
- In this mechanism, fluid movement (mixing) can be visualized as a movement of **portions of various sizes** (**eddies**) that move together.
- **Eddy** is defined as a portion of fluid moving as a unit in a direction often **contrary** to that of the general flow.
- Larger eddies tend to break up forming smaller eddies until they are no longer distinguishable.



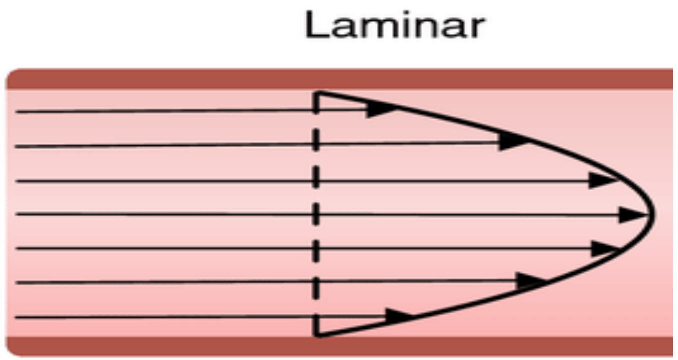


Liquid Mixing Mechanisms

- **Third: Laminar flow (streamline)**
- This type of flow happens when:

1. Mixing **highly viscous fluids**.
2. It can also occur if **stirring** is relatively gentle (**moderate or slow**)
3. May exist adjacent to stationary surfaces in vessels in which the flow is predominantly turbulent.

- This can be described by layers of the fluid moving into another fluid.
- When two dissimilar liquids are mixed through laminar flow, the **shear that is generated** stretches the **interface between them.**
- https://youtu.be/_dbnH-BBSNo?si=L-9q-ohIymEs998J



Liquid Mixing: Laminar Flow

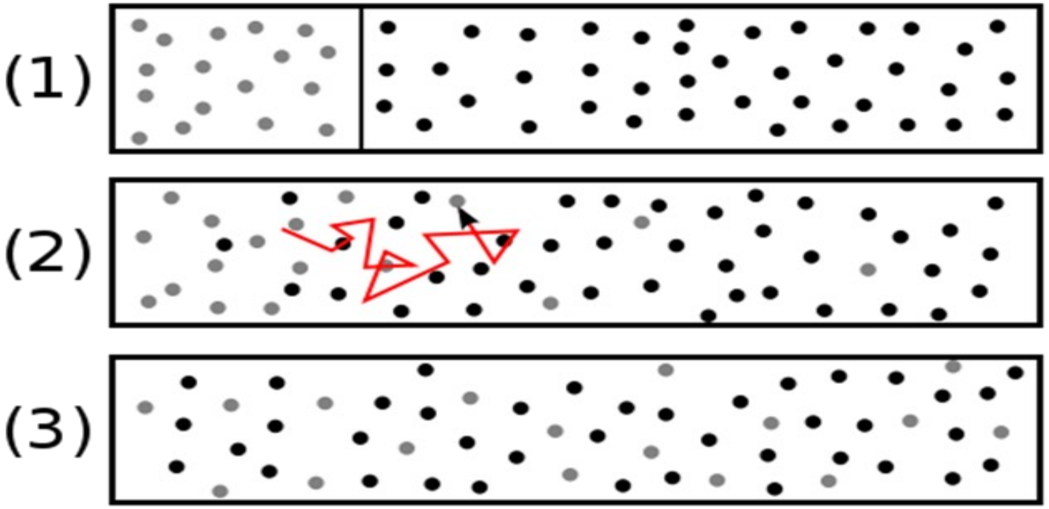
- When 2 dissimilar liquids are mixed through laminar flow, the shear that is generated **stretches** the interface between them. ➔ If the mixer employed **folds** the layers back upon themselves, the number of layers & hence the interfacial area between them **increases exponentially with time**.
  - This relationship is observed **because** the rate of increase in interfacial area with time is proportional to the instantaneous interfacial area.

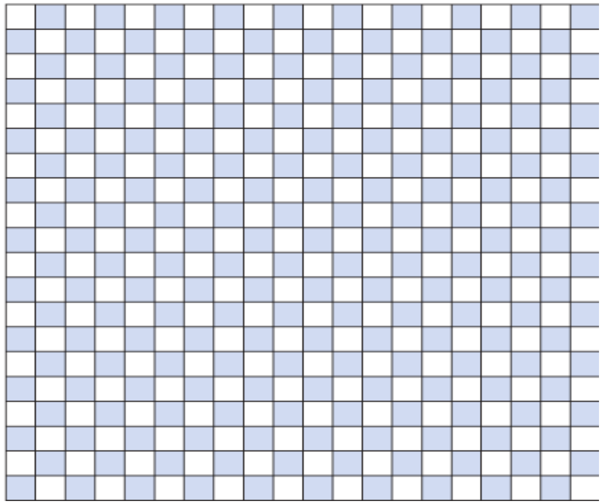
Laminar Flow: Example

- Consider the case where the mixer produces a folding effect & generates a complete fold every 10 seconds.
- Given an initial fluid layer thickness of 10 cm, a thickness reduction by a factor of 10^{-8} is **necessary** to attain layers 1 nm thick (which approximate molecular dimensions ≈1 nm). Since a single fold results in a layer thickness reduction of one-half,➔ **n** folds are required where:
- $(1\backslash 2)^n = 10^{-8}$ ➔ take log of both sides (since $\text{Log}\,(X)^y = y\,\text{Log}\,X$), Log 10=1
- $\log(1/2)^n = n \log ½$ **and** $\log 10^{-8} = -8$ ➔ n log (1/2)= -8 ➔
- n= -8/(log ½) = -8 /-0.3 = 26.6 folds required to get 1 nm
- Since each fold requires 10 seconds ➔ total time required for mixing (equal to **n times 10 seconds)** = 26.6 * 10 = **226 sec or 4.43 minutes**
- **Good mixing** at the molecular level **requires** a significant contribution by molecular diffusion **after** the layers have been reduced to reasonable thickness by laminar flow.

Liquid Mixing: Molecular Diffusion

- **Fourth: Molecular diffusion.**
- Mixing of **molecules** of one fluid through another by random molecular motion.
- The **primary mechanism** responsible for mixing at the molecular level is diffusion resulting from the thermal motion of the molecules when it occurs in conjunction **with laminar flow**.
- In this mechanism, molecules move according to the **concentration gradients** until they result in complete mixing.





Liquid Mixing: Molecular Diffusion

- Molecular diffusion tends to reduce the sharp discontinuities at the interface between the fluid layers and if allowed to proceed for sufficient time result in complete mixing.
- The process described by Fick's 1st law of diffusion (substances will diffuse from areas of high concentration to lower concentration)

$$dm/dt = -DA\, dc/dx$$

- The rate to mass transfer (*dm/dt*) across an interface of area **A** is proportional to the concentration gradient (*dc/dx*) across the interface
- **D** (diffusion coefficient) depends on the viscosity and the size of the diffusing molecules.
- The concentration gradients at the original boundaries are a decreasing function of time, approaching zero as mixing approaches completion.

Mixing Evaluation

•It is important to examine each mixture during the mixing process to ensure that we have enough mixing (i.e. random mixing).

•Danckwerts has suggested **2 criteria** to describe the degree of mixing (quality of randomness or goodness of mixing)

1. **The scale of segregation:**
   - Scale of segregation is expressed in 2 ways either linear or volume scale.
   - The linear scale represents the average value of the diameter of the lumps whereas the volume scale represents the average lump volume.
2. **Intensity of segregation** is a measure of the variation in composition among the various portions of the mixtures.
   - Variation **will be zero** at the end of the mixing

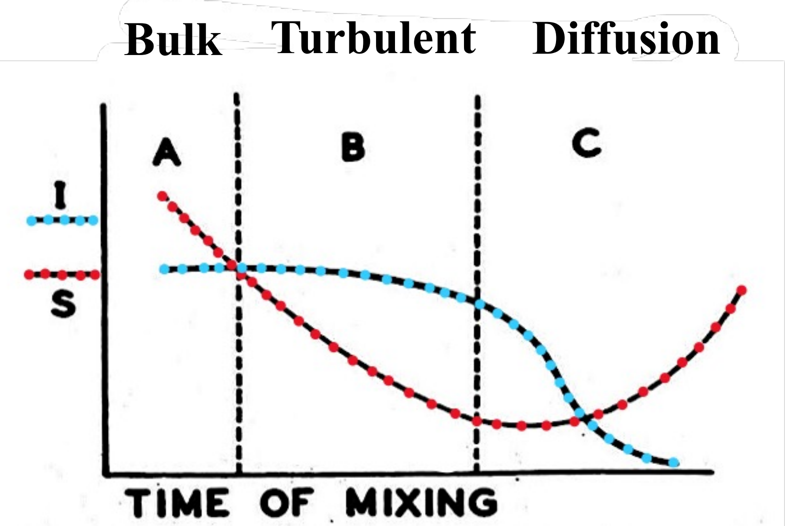


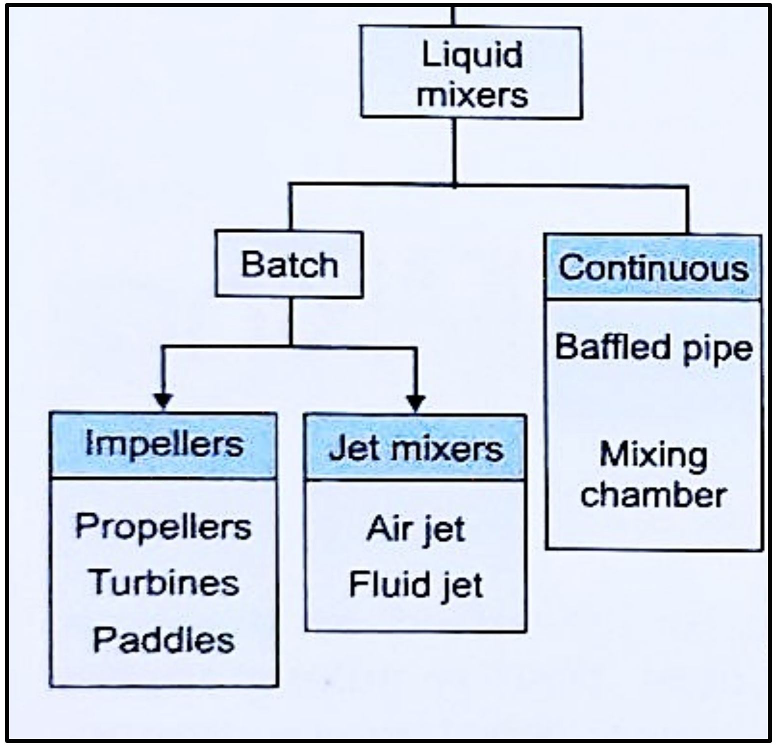
FIG. 1-1. *The intensity of segregation, I, and the scale of segregation, S, as a function of time. Bulk transport, turbulent mixing, and molecular diffusion are predominant over the time periods A, B, and C, respectively. The linear scale of segregation may be seen to increase at the end of the mixing operation. The final mixture will be uniform in composition and may be considered a single lump with a linear scale equal to the linear dimensions of the mixer.*

Equipment for Fluid Mixing

- Generally, fluid mixers consist of a:

1. **Tank or a container** to hold the material being mixed and
2. A suitable **energy transfer tool** such as an impeller, liquid jet, or air stream.
   - Besides supplying power, these tools also serve to direct the flow of material within the vessel.

- **Baffles, vanes, and ducts** are also used to direct the bulk movement of material in such mixers, thereby increasing their efficiency.
- When the material to be mixed is **limited in volume** so that it may be conveniently contained in a suitable mixer, **batch mixing** is usually more feasible.
- However, for **larger** volumes, **continuous mixing** is preferred.



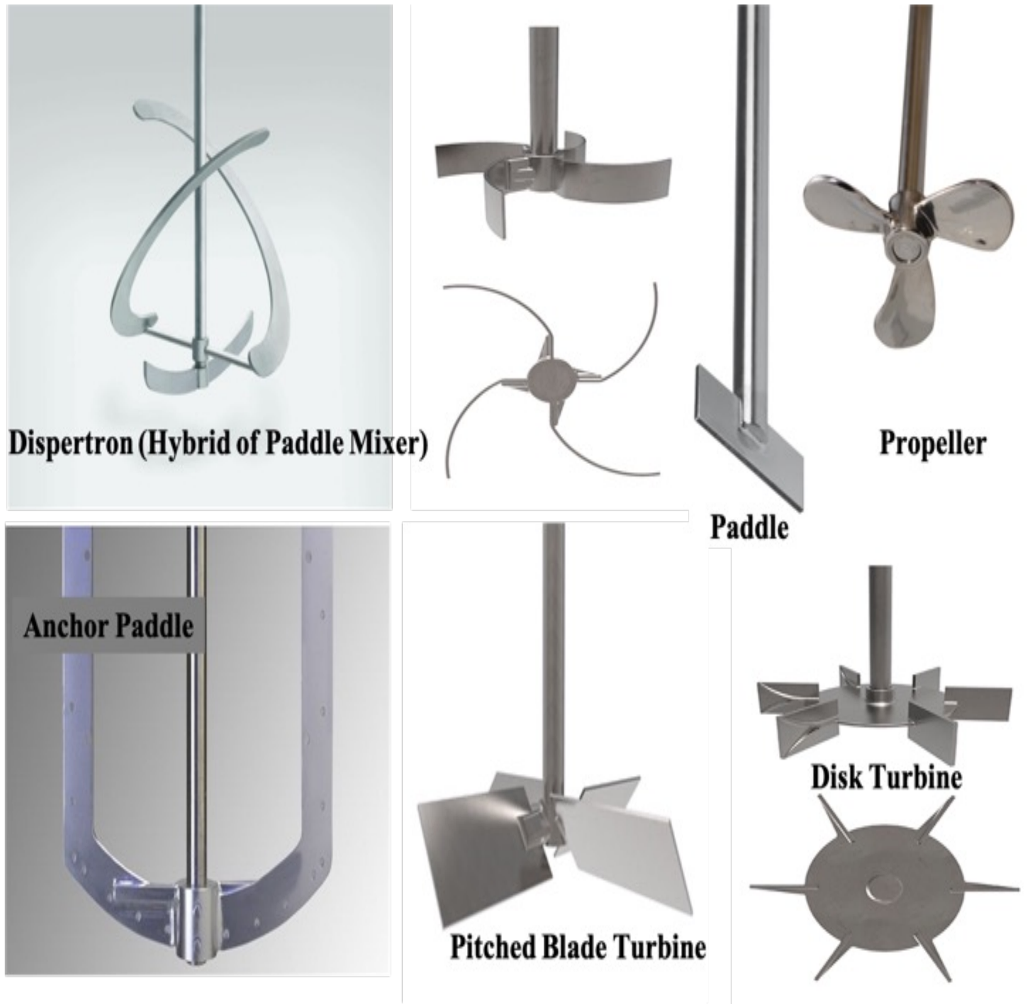
Batch Mixers

- **Batch mixers** are used for small volumes and they are either impellers or jet mixers.

- **Impellers**: these are the **main** mixing units in fluid mixers. It can be either propellers, turbines, or paddles.
- The **distinction** between impeller types is on the basis of:

1. The type of **flow pattern** they produce, or
2. The **shape and pitch** of the blades.

- These impellers cause turbulence and prevent the formation of dead zones.

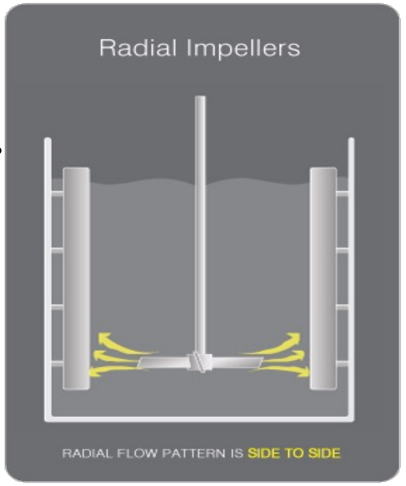


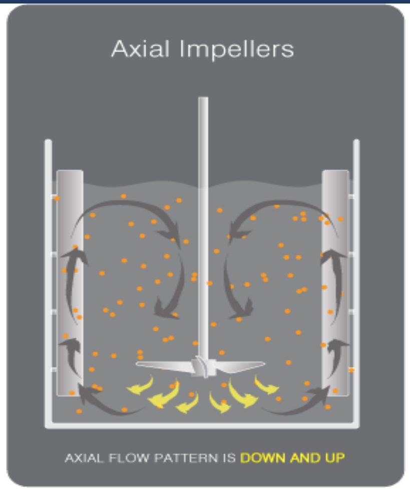
Types of flow in liquid

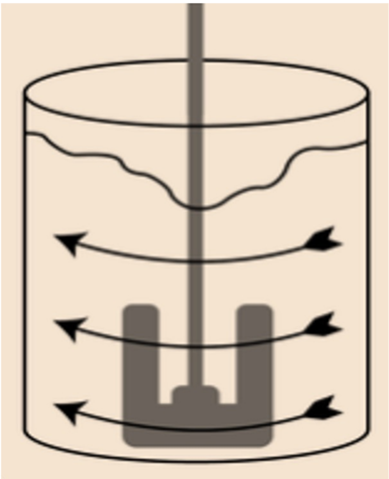
- The impellers **create a current or stream** of liquid that moves in all places of the tank.
- The flow pattern may be analyzed in terms of three components:

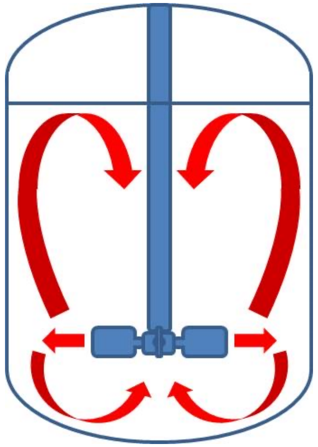
1. **Radial**: perpendicular to the impeller shaft.
2. **Axial or longitudinal (down and up)**: parallel to the impeller shaft.
3. **Tangential**: tangential to the circle of rotation around the impeller shaft.

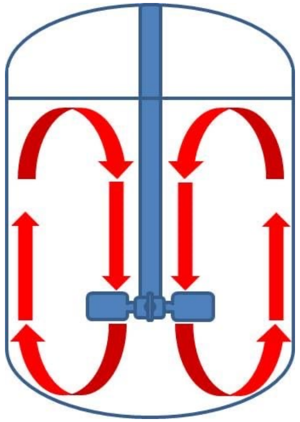
- These may occur singly or in various combinations.

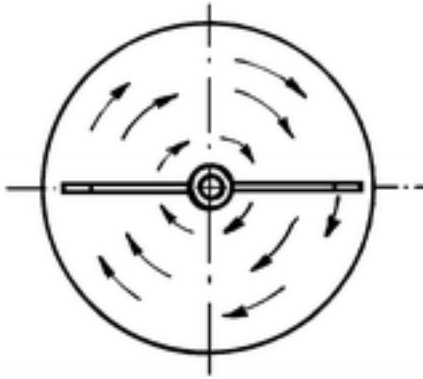


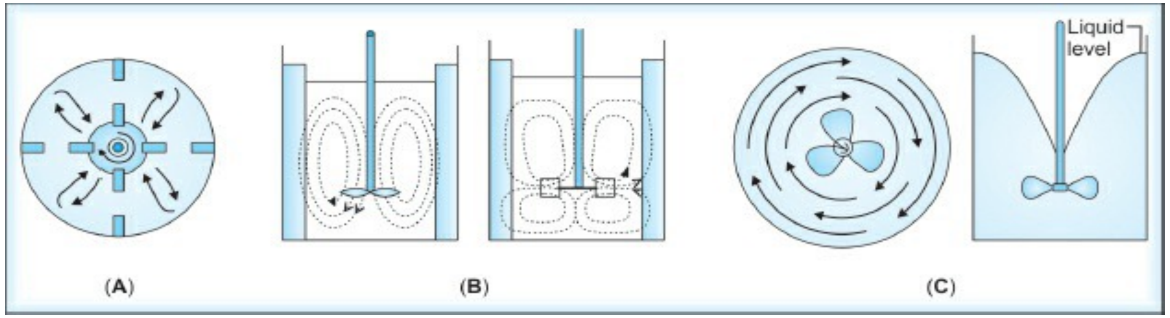






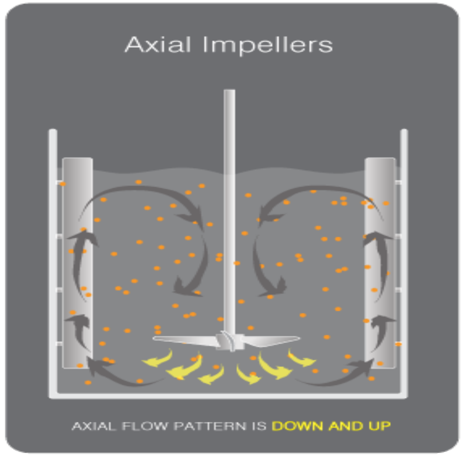


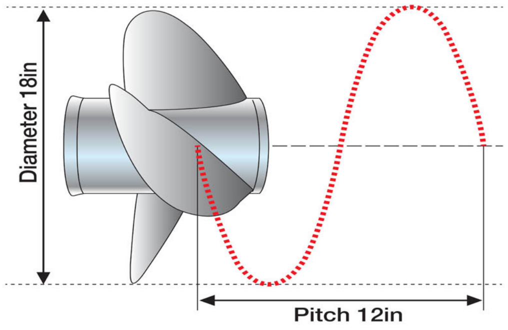




Impeller Batch Mixer: Propellers

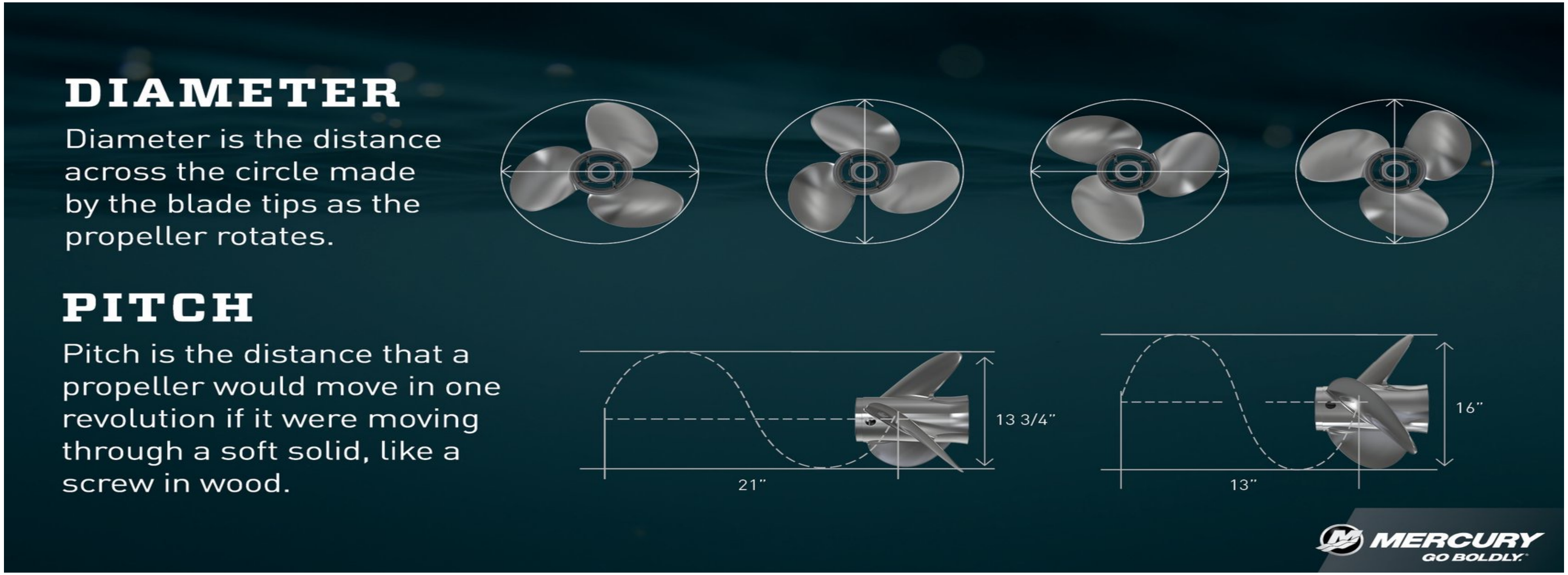
- **Propellers** of various types and forms are used.
- Also, like the machine screws, propellers may be either right- or left-handed depending on **the direction of the slant of their blades**
- The **primary effect** is due to axial flow, **however**, some tangential flow does occur.
  - Also, **intense turbulence** usually occurs in the immediate vicinity of the propeller.
- Propellers are more efficient when they are run at **high speed** in liquids with **low viscosity**.

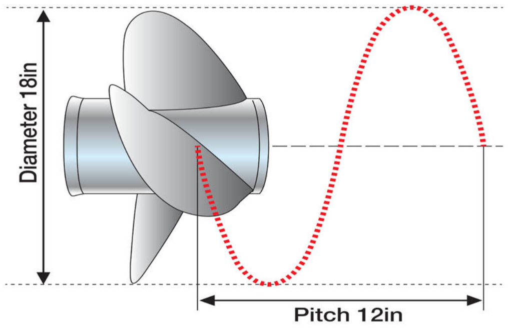




Impeller Batch Mixer: Propellers

- Although **any number** of blades may be used, the **three-blade design** is most commonly used with fluids.
- The blades may be set at any angle or pitch but for most applications, the pitch is approximately equal to the propeller diameter.





Impeller Batch Mixer: Turbines

- They are usually distinguished from propellers in that the blades of the propeller **do not** have a constant pitch throughout their length
- When **radial-tangential** flow is desired, turbines with blades set at a **90-degree** angle to their shaft are employed
  - With these types of impellers, a radial flow is induced by the centrifugal action of the revolving blades
- **If** the turbine blade is **tilted** ➔ it will produce **additional** axial flow **similar to the propeller.**
- Because they lend themselves to a simple and rugged design, these turbines can be operated satisfactorily in fluids **1000 times more viscous** than fluids in which a propeller of comparable size can be used.

- https://youtu.be/kobc3DdaofE

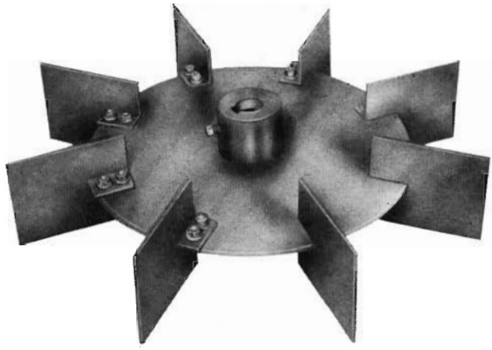
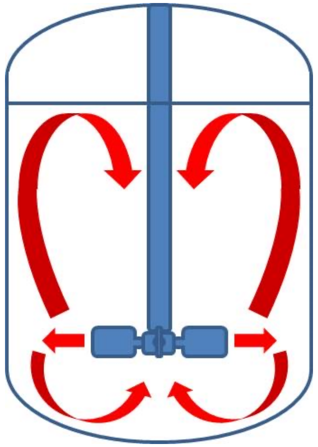
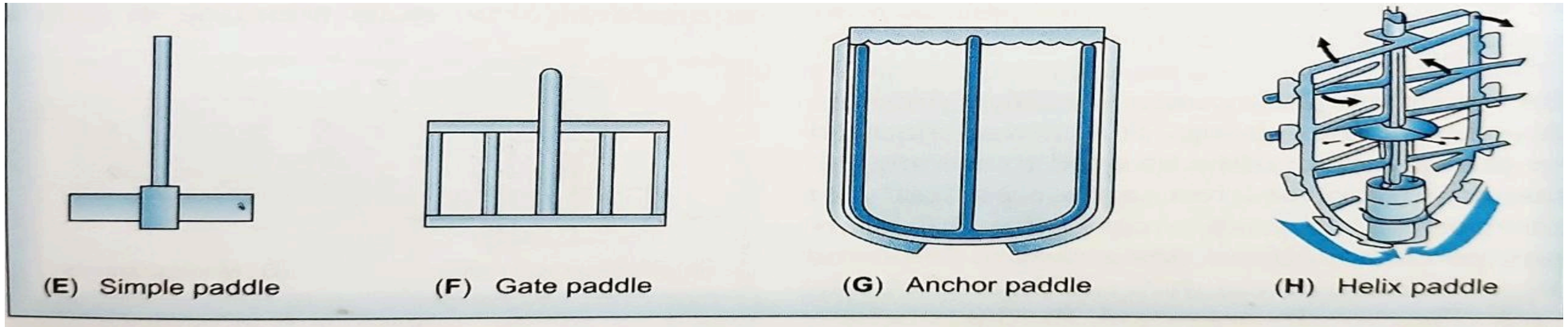


Fig. 2—Flat-blade turbine.



Impeller Batch Mixer: Paddles

- Normally operated at low speed (**lower than 50 rpm**).
- Their **blades have a large surface area** as compared to the tank in which they are employed➔ **they pass close to the tank walls** and effectively mix **viscous** fluids and semisolids which tend to cling to these surfaces.
- Circulation is primarily **tangential**, and consequently**, concentration gradients** (not complete mixing) in the axial and radial directions may persist in this type of mixer **even after** prolonged operation.
- Operating procedures should take these characteristics into account. With such mixers, for example, **ingredients should not be layered** when they are added to the mixing tank.



Figs 1.3A to H: Impeller blade types: (A) Propeller; (B-D) Turbines; (E-H) Paddles

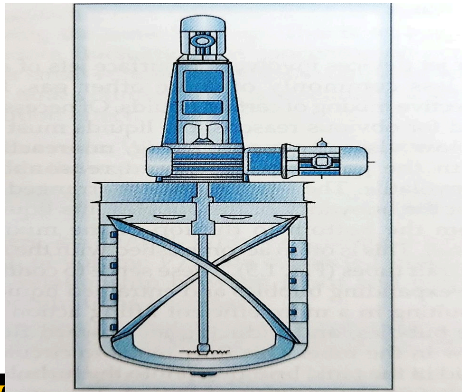


Fig. 1.4: Dispertron: Hybrid of paddle mixer

Batch Mixer: Jet Mixers: 1- Air Jet

- **Air Jets is a** sub-surface stream of air or other gasses used to mix the fluids.
- Fluids should be of:
    1. **Low viscosity**,
    2. **Nonfoaming**,
    3. **Nonreactive** with the gas (or air),
    4. And **reasonably nonvolatile**.
- The jets are usually arranged so that the buoyancy of the bubbles lifts liquids from the **bottom to the top of the mixing vessel**.

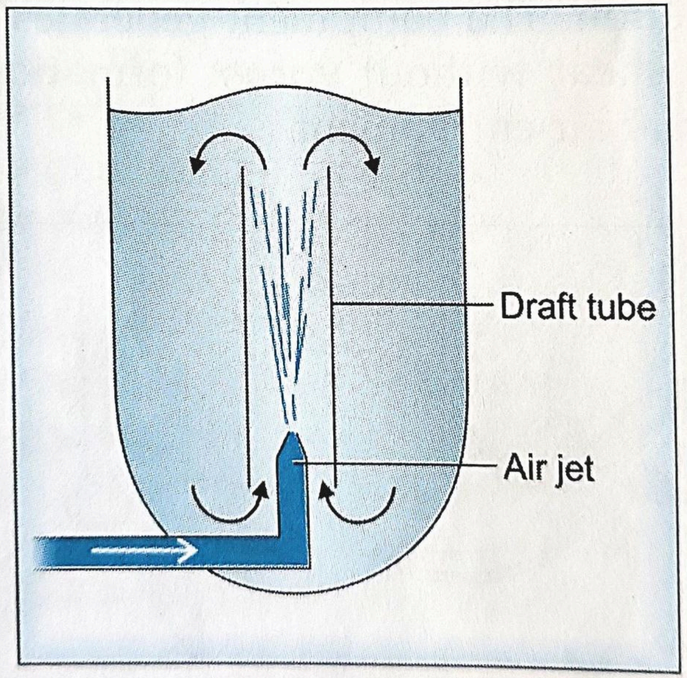


Fig. 1.5: Vertical tank with centrally located air jet and draft tube

Batch Mixer: Jet Mixers: 1- Air Jet

- This is often accomplished with the aid of **draft tubes.**
  - These serve to **1- confine the expanding bubbles** and **2-** entrained liquids,→ resulting in a more efficient lifting action by the bubbles, and inducing an upward fluid flow in the tube.
- This flow tends to circulate fluid in the tank, → bringing it into the turbulent region in the vicinity of the jet.
- The overall circulation in the mixing vessel brings fluid from **all parts** of the tank to the region of the jet itself.
- → Here, the intense turbulence generated by the jet produces intimate (deep) mixing

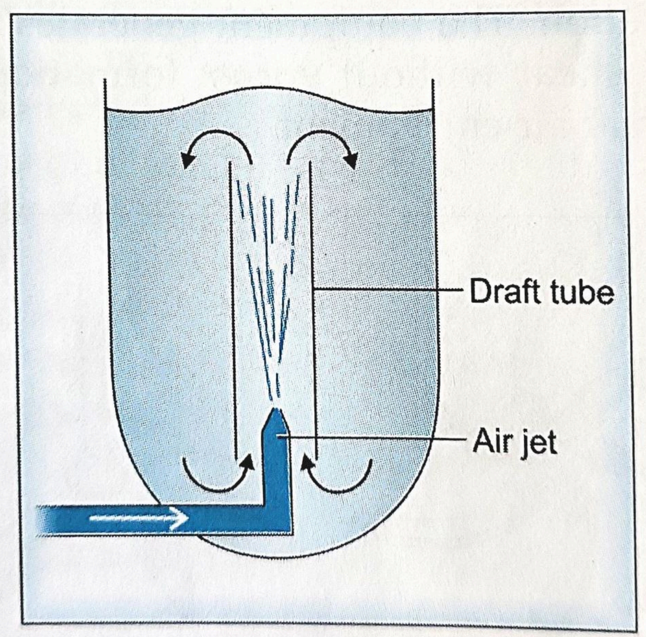


Fig. 1.5: Vertical tank with centrally located air jet a draft tube

Batch Mixer: Jet Mixers: 2- Fluid Jet

- **Fluid jet**: liquid at high pressure is pumped into the tank.
- The power required for pumping can often be used to accomplish the mixing operation, either partially or completely.
- In such a case, the fluids are **pumped through nozzles** arranged to permit a good circulation of material throughout the tank.
- In operation, fluid jets behave **somewhat like propellers** and they generate turbulent flow **axially.**
  - However, they **do not** themselves generate tangential flow, like propellers.
- Jets also may be operated simply by pumping liquid from the tank through the jet back into the tank

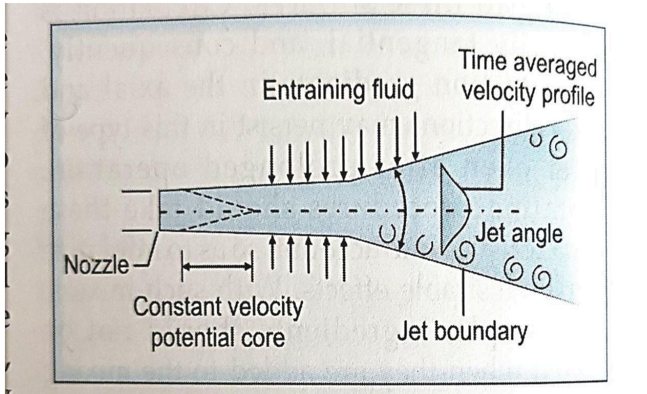
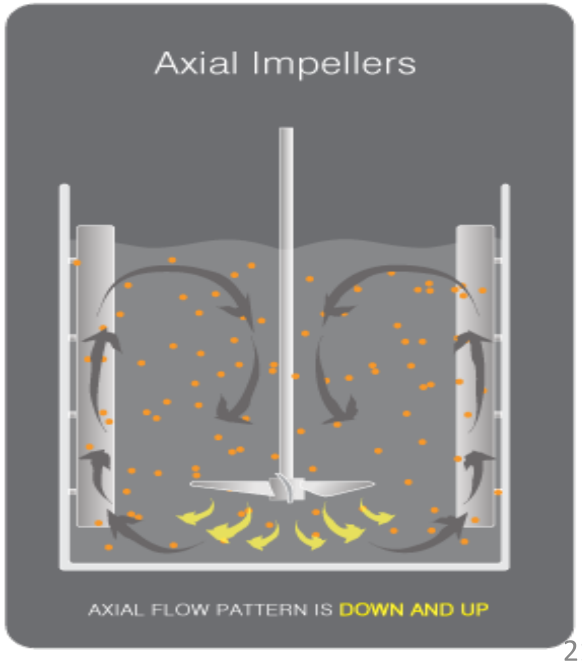
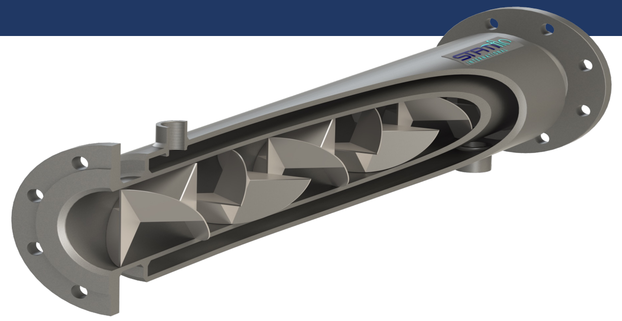


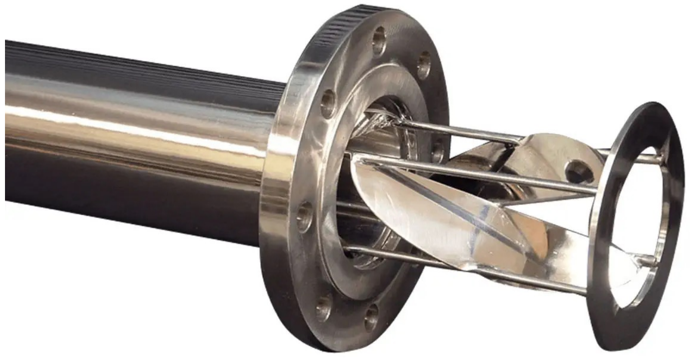
Fig. 1.6: Schematic representation of the fluid jet entering a mass of stationary liquid



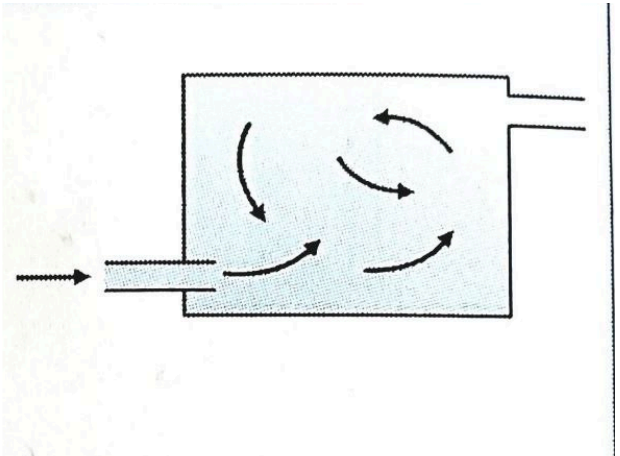
Continuous or In-line Mixers



- **Advantage**: The process of continuous mixing produces an uninterrupted supply of freshly mixed material and is often desirable when **very large volumes** of materials are to be handled.



- It can be accomplished essentially in two ways:
  1. In a tube or pipe (baffled pipe mixers) through which the material flows and in which there is very **little backflow or recirculation**,
  2. Or in a **mixing chamber mixers** in which a considerable amount of holdup and recirculation occurs.



(B) Mixing chamber

- To ensure good mixing efficiencies, devices such as vanes, baffles, screws, grids, or combinations of these are placed in the mixing tube.

Practical Considerations: Vortexing

- **Vortexing**: a vortex develops at the **center of the vessel** when liquids are mixed by **centrally**-mounted vertical shaft impellers.
- This particularly is characteristic of the **turbine** with blades arranged perpendicular to the impeller shaft.
- The **tangential flow** will cause a centrifugal force that may cause vortexing to happen. This is why tangential flow is **not recommended**.
- The tangential flow **will not** result in **any mixing** except possibly near the **tank walls** where shear forces exist.
  - Except in the case of **very low impeller speeds** or at very high liquid viscosities (above 20000 cps). However, these cases are not common in the pharmaceutical industry.

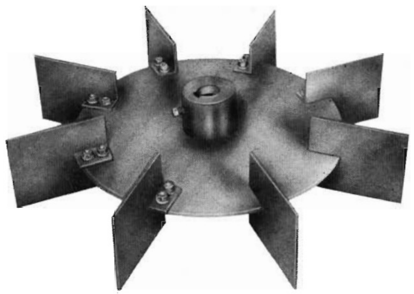


Fig. 2—Flat-blade turbine.

Practical Considerations: Vortexing

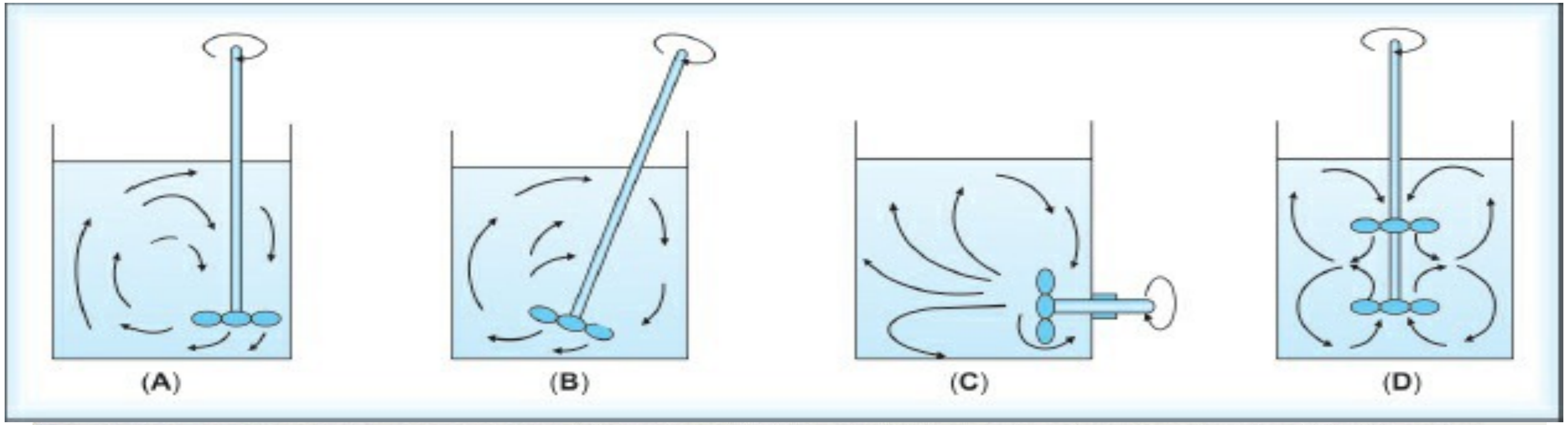
- Vortexing is **not recommended** in mixing due to:
  1. Does not cause real mixing. The full power of the impeller is **not** imparted to the liquid.
  2. Air is drawn onto the impeller and is dispersed into the liquid, which may lead to **foaming**, especially if surfactants are present.
  3. The entrapped air may cause **oxidation** or it can reduce the mixing intensity by reducing the velocity of the impeller relative to the surrounding fluid.

Vortex can be avoided by:

1. Changing the arrangement of the impellers: mounting the impellers off-center or inclined or side position.

2. Changing tank geometry. Asymmetric or angular geometry **but** this will increase the **time** required for mixing since there will be areas in the tank in which the circulation is poor.

3. Using a push-pull propeller: two propellers of **opposite** pitch are mounted on the same shaft to the rotary effects are in opposite directions ➔ and cancel each other.

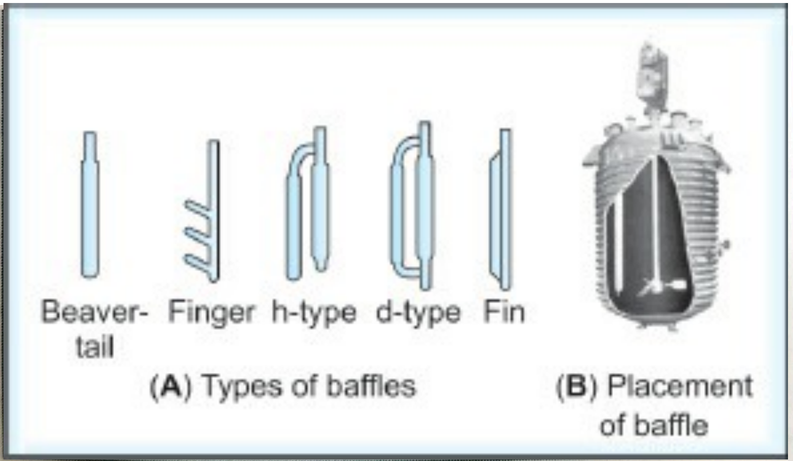


Figs 1.10A to D: Different arrangements of impellers in a vessel with flow pattern to prevent vortex: (A) Off-centre; (B) Inclined; (C) Side-entering; (D) Push-pull propeller

Vortex can be avoided by

4. Using **baffles**: these plates will **convert tangential flow into an axial flow**.

5. **Use of diffuser/stator ring**: that fits around the impeller.



Figs 1.11A and B: Various types of commercially available baffles: (A) Types of baffles; (B) Placement of baffle

- Comparative mixing characteristics of various types of impellers

Parameter	Propellers	Turbines	Paddles
Primary flow pattern	Axial	Radial and tangential, axial (with pitched-blade turbines)	Tangential
Operational speed	High, up to 8,000 rpm	Low, 50–200 rpm	Low, < 100 rpm
Ratio of container-to-impeller blade	Large, ~ 20	Intermediate, ~ 2 to 3	Small, ~ 1.1
Pitch	Not constant	Constant	Constant
Vortex formation	Intensive	Moderate	Does not occur
Application	Positive mixtures: Solutions, elixirs	Negative mixtures: Suspensions, emulsions	Neutral mixtures: Gels, pastes, ointments
Limitations	Suitable only for liquids with low viscosity, < 5 pascal.seconds slurry of 10% solids	Suitable for liquids with moderate viscosity, ~700 pascal.seconds slurry of 60% solids	Suitable for liquids with comparative higher viscosity, > 700 pascal.seconds

Mixer selection

- **Selection will depend on**:

1. **Physical properties** of the materials to be mixed, such as density, viscosity, and miscibility.
2. **Economic considerations** regarding processing for example the **time required** for mixing and the power expenditure necessary.
3. **Cost and maintenance** of the equipment.

- However, the selection of equipment depends **primarily** upon the **viscosity of the liquids** and is made according to the mechanism by which intense shearing forces can best be generated.

Mixer Selection 1- Monophasic System

- **Low Viscosity Systems:**
- Fluids of relatively low viscosity encounter no problems during mixing except when the operational scale is very large.
- Low-viscosity monophasic fluids are **best mixed by the method that**:
  1. Generates a **high degree of turbulence** &
  2. At the same time circulates the entire mass of material.
- These requirements are satisfied by **air jets**, **fluid jets** & various **high-speed** impellers.
- A viscosity of approximately **10 poise** may be considered as a practical **upper limit** for the application of these devices.

Mixer Selection 1- Monophasic System

- **High Viscosity Systems:**
- **Thick creams & ointments &pastes** are of such **high viscosity** that is **difficult** if not impossible to generate turbulence within their bulk & laminar mixing.
- This means molecular diffusion is very important here.
- Mixing may be done with a **turbine of flat blade design**.
  - The power consumption of these devices is **insensitive** to density and/or viscosity.
  - They are a good choice when emulsification or added solids may change these quantities (viscosity and density) during the mixing.

Mixer Selection 2- Polyphasic System

- The mixing **of 2 immiscible** liquids requires the subdivision of one of these phases into **globules**, then distributed throughout the bulk of the fluid.
- These globules are successively broken down into smaller ones.
- Two primary forces play here:
  1. **The interfacial tension** of the globules in the surrounding liquid and
  2. **Forces of shear** within the fluid mass.
- The first force tends to **resist** the distortion of globule shape fragmentation to small globules whereas the opposite with the 2nd force.
- The selection of equipment depends upon the **viscosity** of the liquids and this is made according to the mechanism by which intense shearing forces can best be generated.

Mixer Selection 2- Polyphasic System

- In the case **of low-viscosity systems**, high shear rates are required and produced by passing the fluid under high pressure through small orifices or by bringing it into contact with rapidly moving surfaces.
- **Highly viscous fluids**
- Such as the ones encountered in the production of ointments, are efficiently dispersed by the shearing action of **2 surfaces** in close proximity and **moving at different velocities** with respect to each other.
- This is achieved in **paddle mixers** in which the blades clear out the **walls**.
- These mixers generate shear to reduce globule size and induce sufficient circulation of materials to ensure efficient mixing.

Mixer Selection 2- Polyphasic System

- The mixing of **finely divided solids with a liquid** of low viscosity in the production of a **suspension** depends on the separation of aggregates & distribution of these particles in the fluid.
- This process occurs in a **single mixing operation** provided that shear forces of sufficient to disrupt aggregates.
- As the % of solids is increased or if highly viscous fluids are employed,➔ the solid-liquid system takes on the consistency of a paste or dough. ➔
  - The choice of mixer is either **knead or mull** the materials.
  - **Kneaders** operate by pushing the material by squeezing & deforming them at the same time. Such mixers take several forms.
  - Usually have **counter-rotating blades or heavy arms** that work the plastic mass. Shear forces are generated by the high viscosity of the mass & are effective in the deaggregation & distribution of the solids in the fluid vehicle.