

“Optimizing Mix Performance” prepared by: Ken McCarthy
 Clean Room Coating Solutions
 151 Flint Locke Dr.
 Duxbury, MA 02332
 617-549-4620
kenclnrm@prodiqy.net

Optimizing Mix Performance

This paper presents a discussion of mixing time and its importance to coating operations. Mixing is defined as the reduction in inhomogeneity for a desired process result. Such inhomogeneity can be due to variations in concentration, temperature, or phase. For coating, our objective is on making a uniform product, so our mixing focus will be on uniform concentration. Mixing is used to support a wide variety of processes, but we will concentrate on blending miscible liquids in mix tanks. This paper is complementary to a presentation, “Optimizing Mix Performance”, made at the 2008 AIMCAL Fall Technical Conference in Myrtle Beach, SC, October 19-22, 2008.

A fictitious, but typical, batch sheet is shown below in Figure 1.

Mix 14301, Batch 12	Time Completed Initials
1 Add 1500 L of DI H2O to Vessel D301	_____
2 Set agitator to 30 rpm	_____
3 Set Temp to 42°C, Jacket limit to 60	_____
4 Adjust pH to 5.30+/-0.05	_____
5 Mix for 30 minutes	_____
6 Add 500 L of Polymer ABC solution, Lot 1401	_____
7 Mix for 60 minutes at 60 rpm	_____
8 Add 3 kg of TiO2, Lot 15, slowly over 15 minutes	_____
9 Mix for 60 minutes at 60 rpm	_____
10 Add 1000 L of Polymer XYZ, Lot 98	_____
11 Mix for 60 minutes at 60 rpm	_____
12 Add Premix 578, Lot26	_____
13 Mix for 60 minutes at 60 rpm	_____
14 Cool batch to 30°C	_____
15 Add X-linker J, freshly prepared, Lot ____	_____
16 Mix for 60 minutes at 45 rpm	_____
17 Take sample for solids, ph, and viscosity	_____
18 Continue mixing at 45 rpm while waiting	_____
for analyticals	
Solids _____	
viscosity _____	
pH _____	

Figure 1

Mix times are shown in red. Total batch time is 12 hours, with the bulk of that being mix time.

In coating operations, a bad fluid can not be made good by coating adjustments. On the other hand, in a coating plant, overmixing increases labor costs significantly. Achieving the right balance of effective mixing and just-long-enough mix times is the goal of this discussion. In this paper, measurement techniques to characterize an existing tank, agitator, and fluid will be discussed. Then, a general view of the relationship of mix parameters will be developed. Finally, a calculation method to evaluate the 95% mix time for a new fluid in an existing tank will be shown. The 95% mix time is defined as the point in time where, throughout the mix tank, the concentrations of all materials are within 5% of their completely mixed values.

Measurement Techniques

There are several methods to characterize the mixing performance of a given mix tank, agitator, and fluid, including visual observation, decolorization, sampling, and probe monitors in the tank. Each will be discussed. While there are several variables of interest, in this discussion we will focus on the 95% mix time, sometimes called the blend time. The visual observation gives no indication of mix time, but it is useful for getting a general sense of the mixing effectiveness. The decolorization method does give an estimate of the mix time. Both sampling and the probe monitor methods give a quantitative determination of mix time.

Flow visualization is the first step to take in evaluating the mixing performance in a tank. If the tank is transparent (more likely in a pilot plant set-up), reflective particles can be used with time lapse photography to give some idea of the flow patterns and if there are any dead zones in the tank (areas of little or no fluid movement). The ideal flow pattern is one with lots of varied velocity in all directions. Rapid and efficient mixing will take place in these situations. See Fig.2 below.

If the tank is not transparent (most are not), yet there is good visibility from a manway or other opening, observations can still be made. Good lighting is important to make the best observations. If good lighting is not readily available, a light sheet will help. The light sheet is made up of light sources aligned and directed to form a plane of light to illuminate the flow patterns in the tank. Care must be taken to make sure the light source is secure, so it does not end up in the tank. Any observations made can direct follow-up sample points or measurements. If a model fluid is used, it should be clear and very similar in rheological properties to the fluid of interest. If a model fluid is used, reflective particles can be added to make the flow patterns easier to observe. A photographic or digital recording system will help verify observations after the

experiment is over. If it is not practical to make these visual observations, it is best to proceed to the decolorization method.

The idea of the decolorization method is that it is easier to see flow patterns and areas of stagnation or low flow when color is disappearing than when it is developing. Developing color can block key areas from view. Two acid-base indicators are suggested and an iodine starch indicator system is also discussed. However, any pH indicator system may be used. Bromophenol blue has a deep

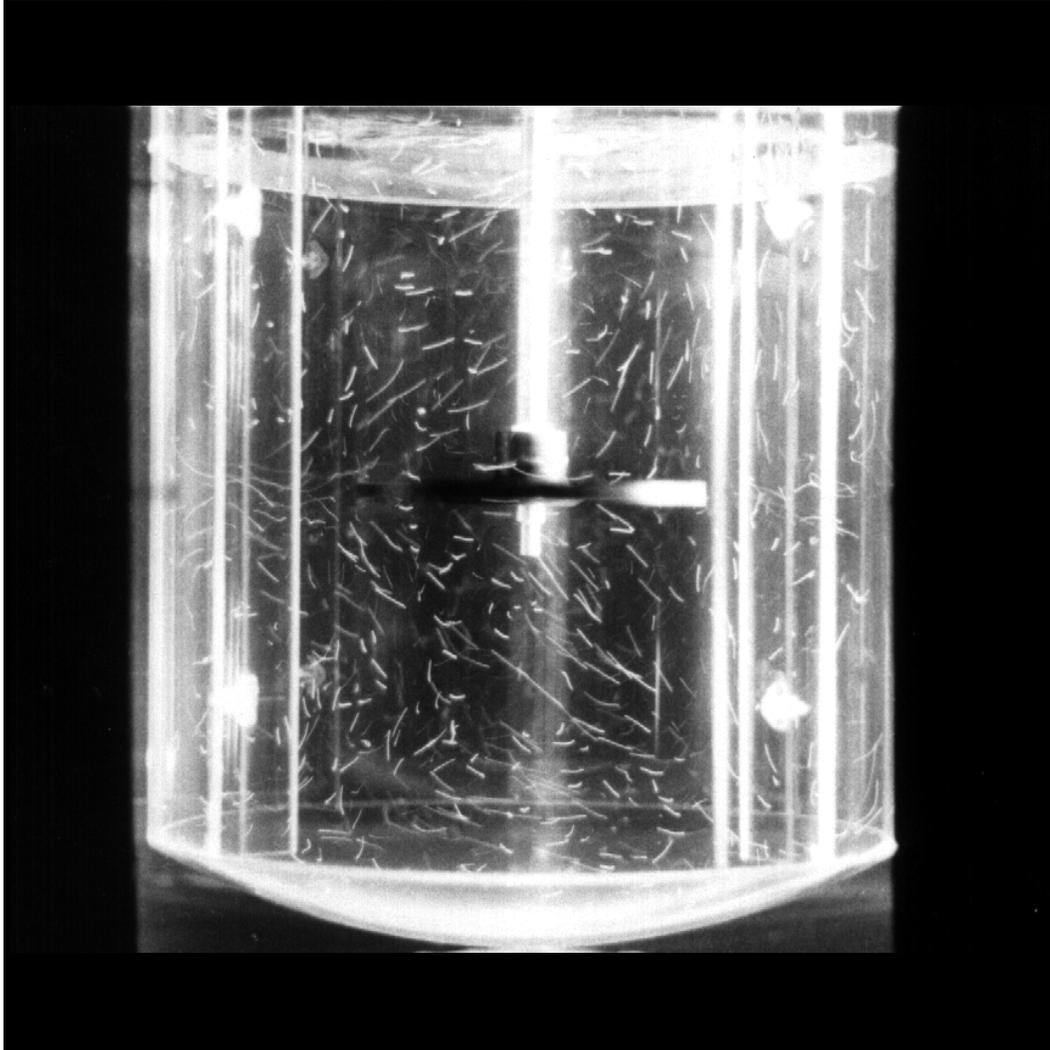


Figure 2 (from Handbook of Industrial Mixing, p.166)

blue-violet color above pH 4.6; it has a yellow color below pH 3.0. For a test, a solution of bromophenol blue and a small amount of base can be added to the tank fluid – the concentration can be determined from a bench test, and scaled up to the tank size. To run the test, sufficient acid needs to be added to bring the pH to below 3.0. Again, a bench test can give a good quantification of the amount of acid needed. The acid should be added in the same way fluids are added

during an actual batch prep. Once the acid is added, observations should start. A recording system can be used to document observations. Areas in the tank where the blue color lingers are the least well-mixed. The estimate for the mix time can be made from the time when the blue color in the tank changes completely to yellow.

For alternatives, another pH system is to use phenolphthalein, which is clear up to pH 8 and changes to red above pH 10. In this system, the colored solution is prepared with phenolphthalein and sufficient base to raise the pH to above 10. At the start of the observations, sufficient acid is added to the mix to lower the pH below 8.0 and then observations are made. A non-pH system uses starch and iodine to develop the color, and thiosulphate to clear the color. As above, observations start when the thiosulphate is added.

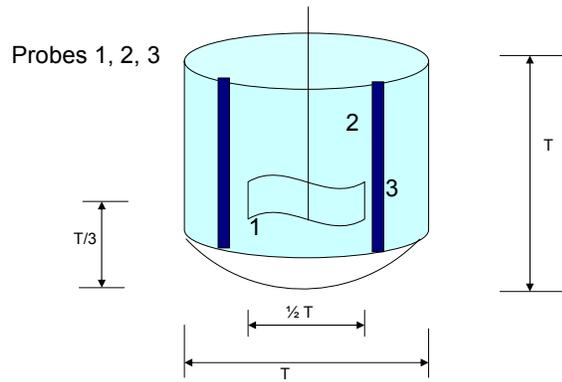
For each of these systems, after an experiment is run, the color can be regenerated by adding the original base, acid, or iodine then starch and iodine. Several experiments can be run in this way, as long as the ionic concentration is below about 0.1M. Higher concentrations can affect the results. For all experiments, observations should note conditions around baffles particularly. Photographic or digital images at regular time intervals are useful for review after the experiment to confirm the time of full clearing. Duplicate runs at a given condition are suggested. Also, easily changed parameters, such as impeller speed, can be done quickly with one batch. A good estimate of the mix time can be made from these experiments. In many cases, this estimate is sufficient.

Two methods are available to provide a quantitative determination of mix time. The first of these is sampling. In this method, at designated times, fluid samples are taken from various points in the tank. This method is difficult to carry out because of the issues in getting secure samples at designated times from various points in a flowing tank. As well, the samples need to be analyzed to get concentration data. If the mix time is short, this fact adds to the difficulty. However, in some cases, sampling is the only option.

The second method, and the one that provides the easiest way to get measured values vs. time if it can be done, is to set up probes in the tank to monitor a parameter such as conductivity or pH. In this method, the position of the probes is critical. One should be located near the sweep of the impeller. A second should be located at about half the depth and about halfway between the impeller sweep and the tank wall. The third should be located right next to a baffle. See Figure 3. The monitors must be chosen so that their shape does not have any significant effect on the flow patterns. In a pH monitoring system, acid or base is added to the tank in a standard way. Then data is collected and recorded well past what may be considered the well mixed point. In a similar fashion, if conductivity is being monitored, an ionic material is added to the tank and data collected. The data from each probe should be normalized as shown in Figure 4

Optimizing Mix Performance

Probe Position Diagram



19

Figure 3

Normalized Measurement Data from Probes

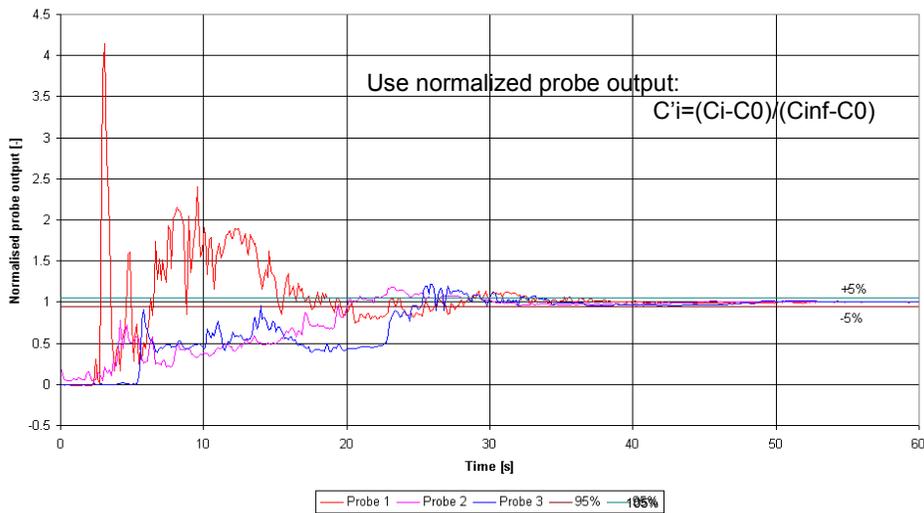


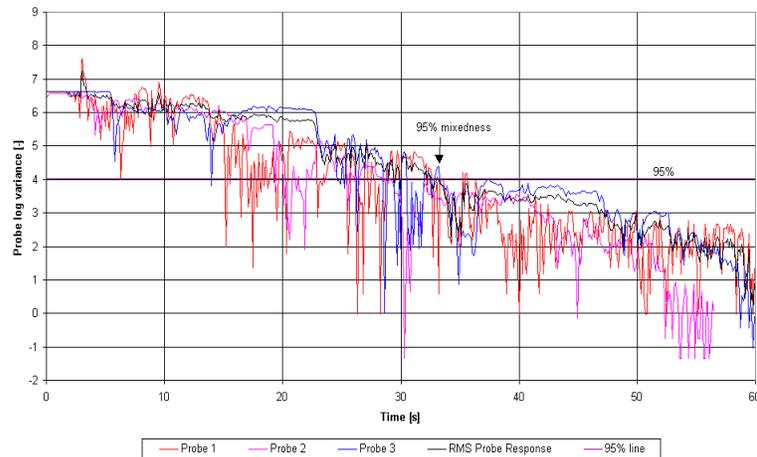
Figure 4-16 (from Handbook of Industrial Mixing, p.173)

20

Figure 4

to eliminate variance in response from probe to probe. The lines in Figure 4 show where the probe response would be at 5% above and below the final concentration. When all normalized values are between these lines, the 95% mix time has been realized. This point can be difficult to determine, but because the probe fluctuations decay exponentially, a plot of $\log \text{var}^2$ vs. time allows for an

Optimizing Mix Performance



$$\log \text{var}^2 = \log(C'_t - 1)^2 \quad \log \text{var}^2_{\text{RMS}} = \log\{1/3[(C'_{t,1} - 1)^2 + (C'_{t,2} - 1)^2 + (C'_{t,3} - 1)^2]\}$$

Fig. 4-17 (from CD accompanying Handbook of Mixing)

21

Figure 5

easier determination of mix time, as shown in Figure 5. The vertical scale in Figure 5 has been adjusted by adding 6.6 to all values.

The mix time for any degree of mixing can be calculated in the following way for a single probe. First, calculate the $\log(\text{var}^2)$ for 95% mixedness ($C' = 0.95$) as $\log(\text{var}^2) = \log(0.95 - 1)^2 = \log(-0.05^2) = \log 0.0025 = -2.602$, adjusted to 4.0 on the graph ordinate. At this point, the mix time is determined to be 33 sec. Since the $\log(\text{var}^2)$ vs time is roughly linear, the mix time for any other degree of mixedness can be determined by the following relationship:

$$\text{mix } t_n / \text{mix } t_{95} = \log[1 - (n/100)] / \log(1 - 0.95)$$

For example, to determine the time for 99% mixedness in the above situation:

$$\text{Mix } t_{99} = 33 \text{ sec} \times \log[1 - 0.99] / \log(0.05) = 33 \text{ sec} \times \log[.01] / \log(0.05) =$$

$$\text{Mix } t_{99} = 33 \text{ sec} \times (-2 / -1.30103) = 51 \text{ sec}$$

Relationship of Mix Parameters

In this section, we discuss the relationship of mix parameters. We review the Impeller Reynolds Number, comparing it to the Reynolds number associated with

flow of liquid in a pipe. Finally, we discuss the significance of Impeller Reynolds Number to mixing phenomena.

At this point, I refer the reader to the CD that accompanies the Handbook of Industrial Mixing, specifically the “Effect of Baffle” video clip. In this video, the effects of having baffles in place is demonstrated after about 10 sec. The flow patterns change from a swirling flow to an axial flow with much better mixing.

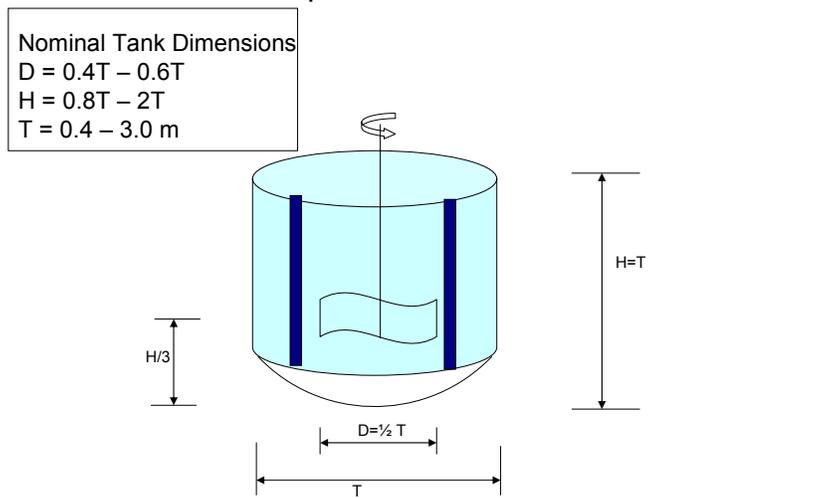
The key figure of merit for mixing is the Impeller Reynolds number. The more well known Reynolds number for flow of liquid in a pipe is calculated as follows: $Re = \rho U d / \mu$ where d is the diameter of the pipe. 0-2000 is considered laminar flow; 2000 – 4000 is considered transitional flow; and >4000 is turbulent flow. The Impeller Reynolds number is characteristic of flow at the tip of the impeller and is calculated as follows:

Imp $Re = ND^2\rho/\mu$ where N is the rotational speed of the impeller, rps; and D is the length of the impeller, m. For the Imp Re , <50 is laminar, 50 – 5000 is transition, and > 5000 is turbulent. Typical values are $N = 1$ rps; $D = 1$ m; $\rho = 1000$ kg/m^3 ; $\mu = 0.01$ Pa-sec (10 cp). For this set of conditions, Imp $Re = 100,000$

Shown in Figure 6 are the ranges of values for key tank geometric dimensions. D is the impeller length; H is the liquid depth; and T is the tank diameter.

Optimizing Mix Performance

Relationship of Mix Parameters



26

Figure 6

Grenville has evaluated many experiments over a broad range of conditions, including impeller shape, length, and speed, tank size and tank level, utilizing the

geometries shown in Figure 6. He has developed the following relationships to connect all this data in the turbulent region and in the transition region. For the turbulent region, the relationship is:

$$(P_o)^{1/3} N [\text{mix time}] D^2 / (T^{1.5} H^{0.5}) = 5.2 \quad (1\sigma = \pm 10\%)$$

where P_o is the Impeller Power Number, unique to each type of impeller

From this relationship, mix time can be calculated if the other variables are known. In addition to the above geometric relationships, baffles are $\sim 0.1T$ and the impeller is located at $1/3H$ above the low point in the tank.

For the transitional region, the relationship is:

$$(P_o)^{2/3} N [\text{mix time}] \text{Re}(D^2/T^2) = 33,489 \quad (1\sigma = \pm 17.4\%)$$

for many combinations of impeller shape, impeller speed, impeller size, tank size, and tank level. Because the Impeller Reynolds number, and hence viscosity, is part of this relationship, non-Newtonian fluids need special attention in this region. Such fluids are beyond the scope of this paper.

New Fluid in an Existing Tank

A demonstration of how these relationships can be used to calculate mix time follows. A new fluid is proposed for an existing tank. The new fluid has a density (ρ) of 980 kg/m^3 and a viscosity of 100 cp or 0.1 Pa-sec . In the tank, the impeller length (D) is 1 m , and a typical speed (N) is 1 rps . The first step is to calculate the Impeller Reynolds number: $\text{Re} = ND^2\rho/\mu = 1 \times 1^2 \times 980 / 0.1 = 9800$ - turbulent region

$$P_o^{1/3} N [\text{mix time}] D^2 / (T^{1.5} H^{0.5}) = 5.2$$

For P_o of 1.8 , N of 1 rps , D of $0.5T$, $H=T$, we get:

$$\begin{aligned} [\text{mix time}] &= 5.2 / [1.8^{1/3} \cdot 1 \cdot ((0.5T)^2 / T^2)] \\ &= 5.2 / [1.2 \cdot 1 \cdot 0.25] = 5.2 / 0.3 \\ &= 17 \text{ sec} \end{aligned}$$

For design, use 3σ limit of constant, $5.2 \rightarrow 6.8$, and

$$[\text{mix time}] = 23 \text{ sec}$$

Conclusions

There are several conclusions that can be made from an analysis of the Grenville Correlation:

All impellers of the same diameter are equally energy efficient (same mix time at the same power per unit mass of fluid).

A larger impeller diameter will achieve a shorter mix time for the same power input per unit mass.

Mix time is independent of the fluid's physical properties in the turbulent regime. When scaling up at constant power per unit mass and constant geometric ratios, mix time will increase by the [scale factor]^{2/3}.

(Handbook of industrial Mixing, p.511)

My overall conclusions for this paper are as follows:

Optimal mixing is a vital part of coating operations. In this paper, measurement techniques to characterize an existing tank and agitator have been discussed. A general view of the relationship of mix parameters has been shown. Finally, a calculation method to evaluate a new fluid in an existing tank has been demonstrated. Balancing batch sheet mix times with calculated or measured mix times is a question that must be answered for each individual case. This analysis demonstrates shorter mix times are worth considering.

Acknowledgement

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