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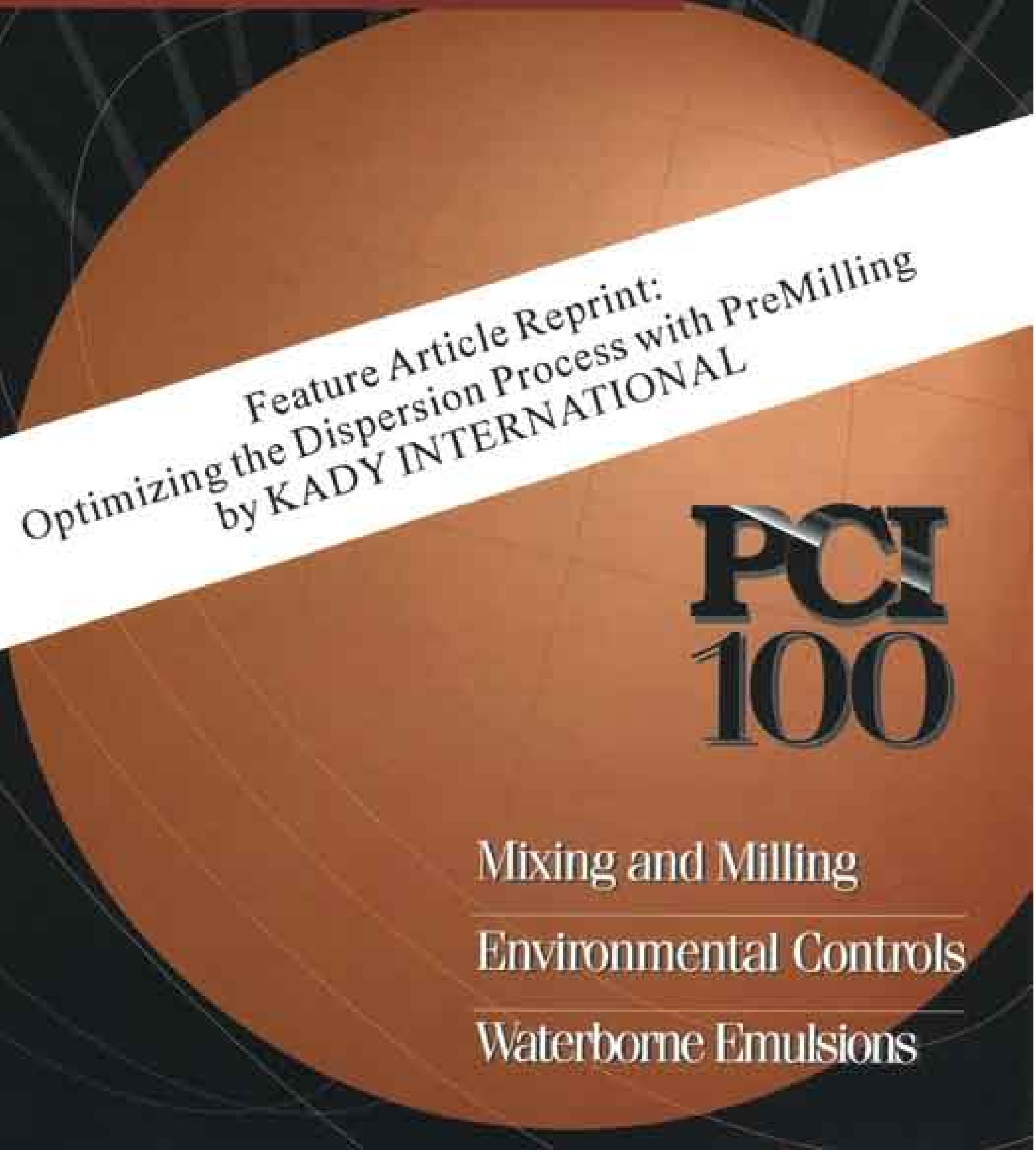


Feature Article Reprint:
Optimizing the Dispersion Process with PreMilling
by KADY INTERNATIONAL

PCI 100

Mixing and Milling

Environmental Controls

Waterborne Emulsions

By David Ulrich Midwest Regional Sales Manager,
James Schak Northeast Regional Sales Manager
and Robert Kritzer Vice President Kady International Scarborough, Maine

Premilling Can Optimize Your Dispersion Process

This article focuses on premixing and also discusses re-engineering the complete milling line.

Coatings requirements have dictated higher quality and finer dispersions. Producers have added more media mills that have multiple passes to obtain these finer grinds. However, using multiple passes and slow flow rates has created inefficient manufacturing. Too much attention has been given to the final milling stages and not enough to the complete process. This article will examine the premix step to re-engineer the complete milling line.

The technique called "premilling" combines the premix step with the early stages of milling. Delivering a finer dispersion to the media mill not only reduces the final milling load, but also allows the use of smaller, more efficient media. The residence time and the number of passes can be reduced and can still achieve a better final grind.

Milling Processes

In the past, coatings production has been viewed as a three-stage approach.

• **Premixing.** Various wet and dry ingredients are blended together. In a well-controlled process, some minimum grind on the Hegman grind gauge is achieved, but it is more common to run for a predetermined amount of time, such as 30 minutes.

• **Milling.** The pigments are mechanically deagglomerated until a final particle size is reached.

• **Letdown.** Various additives are introduced along with any vehicle that was held out during premixing.

The premixing stage has been looked at as a necessary evil before the real work can take place. High-speed dispersers are normally chosen, although, in some cases, low-speed blade mixers are still used. For the most part, producers are still operating in the dark ages with premixing.

On the other hand, the final milling stage has received much more attention. The industry standard 40 years ago was sand mills. These required long residence time and multiple passes. The next step was to replace the sand mill with more effective media, such as ceramic beads or balls. These vertical mills were then replaced with the horizontal small media mills.

In the last few years, these mills have become more efficient with the development of faster shaft speeds, higher media loads, more discs and longer chambers. Further design enhancements will probably only bring marginal results.

The letdown stage is a blending operation usually handled with a blade or propeller mixer. This requires only distributive mixing of the already dispersed pigment, additional vehicle and/or additive.

Dispersion Steps

An evaluation of dispersion equipment efficiency requires an understanding of what is needed to obtain the final result. The three steps in achieving a high-quality dispersion are:

• **Wetting.** The dispersion process must first remove the air and moisture inside the pigment and replace it with the vehicle.

• **Deagglomeration.** Agglomerates are formed either as the pigment was manufactured, or at the wetting stage.

• **Stabilization.** The particles, after they are wetted and uniformly dispersed, must be stabilized so that they do not reaggregate. This is accomplished by introducing an opposite electrical charge to negate attraction. Surfactants, or binders, aid in stabilization.

Dispersion Equipment Design

The wetting and deagglomeration of the pigment is obtained by exerting mechanical energy into the product and overcoming the forces that bond the particles together. The amount of force required is a function of the final particle size desired and the characteristics of the matrix material.

The particle bonds can be a result of several factors: unbalanced surface charges at the crystal faces; Dipole fields (similar to magnetic fields) on the crystal; direct cementation of crystals through reaction by-products or impurities or resins; and mechanical interlocking formed by the pigment manufacturing drying process.

The transference of bond-breaking energy is through impact, attrition shear or compression. Equipment examples that use these forces are enclosed impeller, open impeller, kneader, roll mills and media mills. The kneader and the roll mill are designed for high viscosity and will not be addressed in this article.

There is no singular piece of equipment capable of handling every milling application efficiently. There are too many factors that affect the dispersion process to allow uniform processing. These factors include viscosity, vehicle, pigment hardness, surface charges, final grind requirements, solids content, color strength, gloss, aeration tendencies, temperature limitations and formulation.

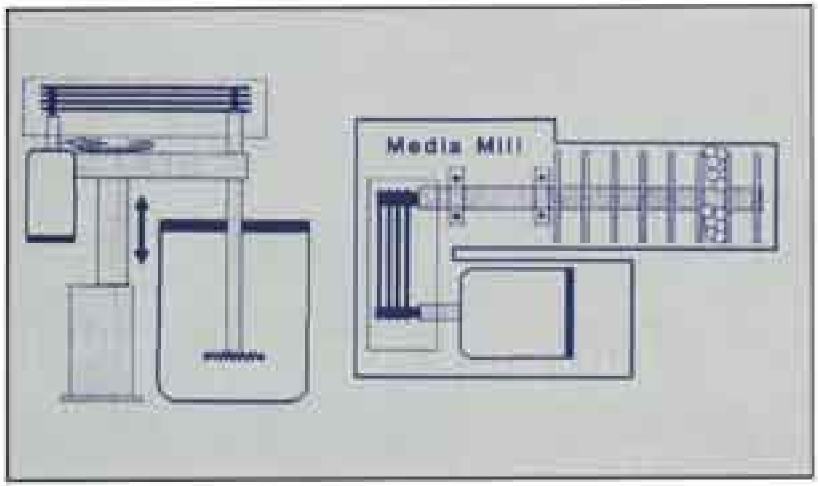
The three pieces of equipment used today to handle the spectrum of pigment dispersions are as follow.

• **High-speed blade dispersers (or open impellers).** This distributive mixing device is good for initial pigment wetting at the premix stage. This unit relies on mechanical shear as its primary input, with a secondary force of attrition.

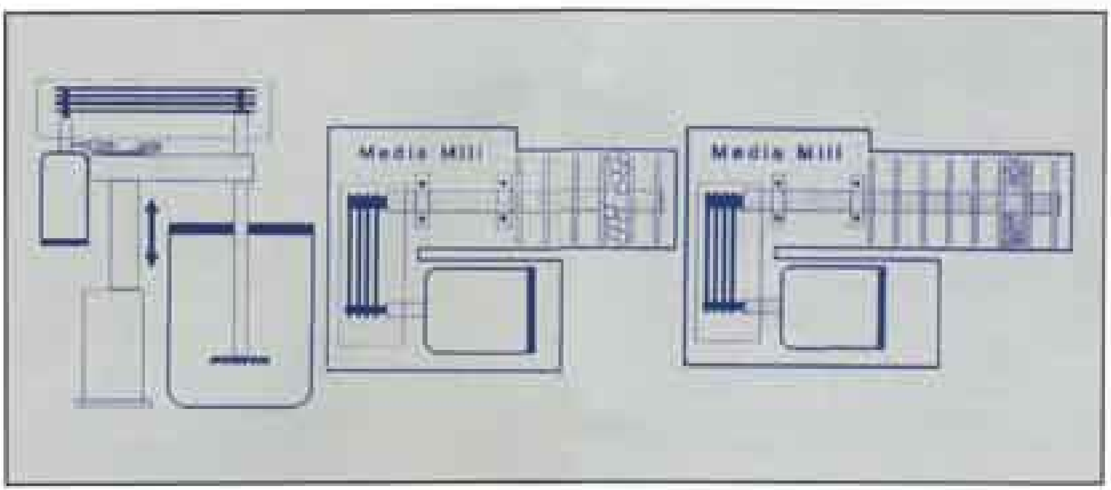
• **Rotor/stator mills (enclosed impeller).** This dispersive mixing device is good for premix, premilling and softer pigment grinds. This design uses impact and attrition with shear compression as a secondary source.

FIGURE 1

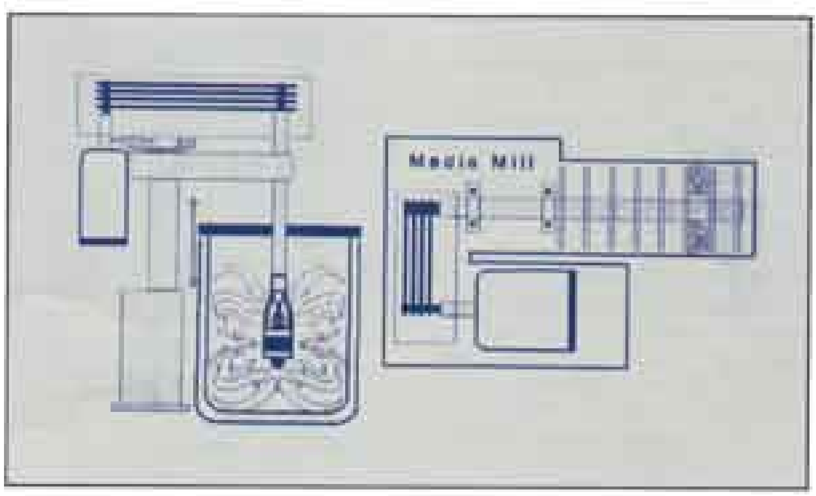
System #1
High-speed disperser followed by a horizontal media mill.



System #2
High-speed disperser followed by a large media mill followed by a small media mill.



System #3
Rotor/stator mill (9000f/m rotor tip speed) followed by a small media mill.



• **Horizontal media mills.** This dispersive mixing device is designed for achieving fine grinds of hard pigments. The work is done through a combination of shear and impact with attrition as a secondary force. This article will focus on applications that require media mills for grinding hard pigments to a 7.5 Hegman (The industry uses the term “grinding” to describe the breakdown of agglomerates). To choose the most effective dispersion system, look at the design of the horizontal media mill to optimize the complete process.

Small Media Mill Design

Media mills operate by inducing impact from the collision of media with itself and the vessel walls. Shear is introduced as adjacent media particles pass by one another. They are most effective over a small particle size range reduction. This is due to the important relationship between media and agglomerate size. The rule of thumb is that the most efficient media diameter needs to be around 10 times the size of the initial largest pigment, according to Leo Dombrowski’s article, “Optimizing Small Media Mills,” *(American Paint & Coatings Journal)*, November 29, 1982 and December 6, 1982.

If the agglomerates are too large, they tend to act as a piece of media, which effectively eliminates shear as a dispersion tool. The role of impact is also reduced since the media particles have lost some of their size and weight advantage. Attrition now becomes the major contributor to any work being done. Because of its media size and shaft speed, the media mill is simply not well suited for attrition.

If the agglomerates are too small, they tend to get lost in the mill. Impact becomes ineffective because there are not enough media balls to do the job. This is due to an increase in the number of total particles during the dispersion process. The number of total particles will be increased by a factor of eight by grinding its original size in half (0 Hegmen to 4 Hegmen). In this case, attrition is all but eliminated, leaving only shear as the means of dispersion.

Fig 2 **Media/Largest Particle**

Media Mill Diameter (Media-microns)	Largest Particle (microns)	Ratio (Media/Pigment Particle)
1,000 (1mm)	100 (0 Hegman)	10/1
1,000	89 (1 Hegman)	11/1
1,000	50 (4 Hegman)	20/1
1,000	25 (6 Hegman)	40/1
1,000	13 (7 Hegman)	77/1
1,000	6.4 (7.5 Hegman)	156/1
500 (0.5 mm)	100 (0 Hegman)	5/1
500	89 (4Hegman)	8.5/1
500	50 (4 Hegman)	10/1
500	25 (6 Hegman)	20/1
500	13 (7 Hegman)	38/1
500	6.4 (7.5 Hegman)	78/1
250 (0.25 mm)	100 (0 Hegman)	2.5/1
250	89 (4 Hegman)	2.8/1
250	50 (4 Hegman)	5/1
250	25 (6 Hegman)	10/1
250	13 (7 Hegman)	19/1
250	6.4 (7.5 Hegman)	39/1

A widely used media mill production setup (see Figure 1) utilizes a high-speed blade premixer to be “on scale” (0 Hegmen) or at about 100 microns. A media mill using 1-mm (1,000 microns) beads (a range of diameter sizes is usually denoted) is used to achieve the final grind of 7.5 Hegmen (6.4 microns).

The 1-mm media mill usually requires two passes because of the large particle size reduction of 100 microns, down to 6.4 microns. As Figure 2 shows, this setup starts out fine, with the required 10: 1 ratio (1,000 microns per 100 microns) on the first pass, but on the second pass, this ratio jumps to an inefficient 50: 1 (1,000: 20).

To maintain milling efficiency, one must choose the media by the final grind requirements, not the initial particle size. The product then must be premilled to deliver particles within the 10: 1 range. A rule for choosing the media diameter is not well defined, but a four-fold reduction usually gives an efficient design without sacrificing production rate our without requiring multiple passes. Of course, this four-fold reduction will be disputed, since it depends on a number of variables. Testing is always recommended.

A media diameter can be arrived at by multiplying the four-fold reduction by the 10: 1 ratio to get a 40-fold multiplier of the final particle size requirement. Looking at the 6.4-micron final grind, multiply this number by 40 to get 256 microns, or a 0.25-mm (250 micron) bead.

The next step is to work backwards to find that the product must be premilled to a 6 Hegman (25 microns – 250: 10) to meet the 10: 1 ratio. Next, choose the equipment necessary to handle the premilling portion of the process. Some companies have used a second media mill with large (1-mm) media with good success (see Figure 1).

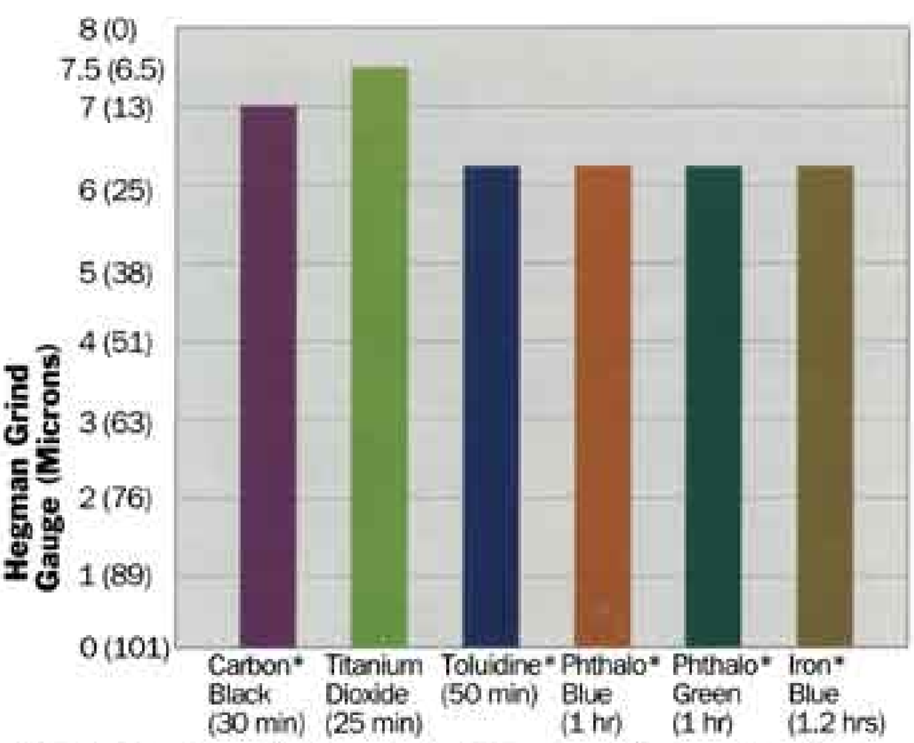
In this case, a high-speed disperser produces a premix of 100 microns (0 Hegman). The first mill grinds the premix to Hegman 6 or better, and the second mill will finish the grind. The downside of this approach is the additional cleanup required, as well as the high capital cost. The best approach would be to use a piece of equipment that can handle both the premix and premilling requirements efficiently, such as a rotor/stator mill (see Figure 1 on page 2).

Rotor/Stator Mills

Rotor/stator mills have been successfully used to achieve a final grind on softer pigments like whites and yellows. Recently, it has been used to achieve a finer premix. This is the concept of premilling. High-speed blade mixers are a common premixing device, but they lack the dispersing power of high-speed rotor/stator mills that operate at 9,000-feet-per-minute rotor tip speed.

Fig. 3 **Hegman Grind Gauge for Rotor/Stator Mill**

Typical Dispersion Time for Rotor/Stator Mill (9,000 ft/m)



*Note: The final grind capability depends on formulation. Some of these hard pigments are difficult to reach a 1-4 Hegman on a rotor/stator mill.

The rotor/stator has the capability to achieve a 6 or 6.5 Hegman (see Figure 3) on some hard pigments. This is an ideal range for a small, horizontal media mill to be efficient in getting down to the 1- to 10-micron range. This was confirmed by Malcolm MacKay and Isadore Rubin in a recent article examining a phthalocyanine blue ("Methods to Obtain Dispersion and Grinding," American Pain & Coatings Journal, September 22, 1993).

MacKay and Rubin compared the performance of 2- to 2.5-mm, 1.2- to 1.6-mm and 0.8- to 1-mm media in a horizontal media mill with a rotor/stator premix. The results indicated that the better premix allowed use of smaller media in the media mill and reduced the number of passes from two to one without affecting dispersion time.

MacKay and Rubin used a rotor/stator mixer with a rotor tip speed of only 5,000 feet per minute for their testing. A rotor/stator mill with much higher rotor tip speeds would allow an even finer media to be used. The particle size leaving the rotor/stator mixer was not recorded in this article. Figure 4 reflects a three-hour production time savings of eliminating one pass on the media mill.

The advantage of a rotor/stator mill is to handle the premix step and induce a tremendous amount of dispersing power in a short period of time for premilling. There are a number of rotor/stator manufacturers with various designs. The critical design elements are the 9,000-foot-per-minute rotor tip speed and a slotted rotor to transmit the full impact of the dispersing power available.

Heat generation is an important point when using rotor/stators or media mills. Cooling water is usually required to maintain the optimum process temperature. The amount of heat generated can be calculated by doing a heat balance around the processing unit. The energy is imparted through the drive motor at a rate of 2,547 btus horsepower.

Heat Balance

For example, to calculate the temperature rise of a 200-gallon batch with a 100-hp mill operating at full amp load, assume the worst-case scenario that 100% of the mechanical energy is transmitted to the mix. Typical dispersers transmit around 70% to 90% of the mechanical energy into heat, depending on the vehicle, heat capacities, pigment and viscosity.

It is assumed that the specific heat equals 0.5 btu/F° and that specific weight equals 8.34 pounds per gallon. Also, assume the following.

Q(heat input-motor)=M(weight in pounds) * specific heat (Cp)* Delta T

(2,547 btus per hour)=(200 gal. * 8.34 lbs. per gal)(0.5) (Delta T)

152° F= Delta T in 30 minutes (if cooling was used)

5° F= Delta T in one minute

Figure 5 depicts a comparison of the three different milling scenarios previously discussed: high-speed disperser followed by a horizontal mill with large media; high-speed disperser followed by a horizontal mill with large media, then by a horizontal mill with small media; and rotor/stator mill followed by a horizontal mill with small media.

The fastest processing line is System 2 with two media mills, but it is also the most expensive and the hardest to clean. The least expensive system is System 1 with a two-pass media mill, but it takes about twice the time. System 1 is trying to reduce particle size from 100 to 6.5 microns (15.6-fold reduction). This is asking a media mill to do too much with too large media. The result is two or more passes at low production rates.

Considering system price, production time and cleanup, it is apparent that System 3, the rotor/stator (small media mill) is the most efficient approach. It combines the premix and premilling stages into one unit without additional equipment. The finer dispersion fed to the small media mill is in the 10: 1 target for optimum efficiency. The actual particle size reduction is from 25 to 6.4 microns (3.9-fold reduction), which can be handled in one pass with 0.25-mm media at a high production rate (0.25-mm media is not the most common, but there are some color houses and ink companies using it). Dombrowski reported that "the premix was important if high output is to be seen from the small media mill."

Selecting Equipment

The basic logic for low-viscosity dispersion equipment selection is to choose the simplest, most efficient system. The first attempt should be to try to use a blade mixer or even a prop mixer. This is the least expensive method and has all the processing done in one tank.

If this is unsuccessful, the next step is the rotor/stator mill. Again, it has the advantage of one-step processing. If all else fails, the media mill should be tried. The disadvantages of the media mill are as follows.

- The unit needs a premix tank.
- The media mill is a low-rate, continuous device, so it takes a number of hours to process the batch.
- The unit is difficult to clean.
- The media mill is expensive in relation to is production capacity.

To avoid multiple media mill passes, try a rotor/stator mill as a premilling device.

Premilling Steps

1. *Choose the final media size by the final grind requirements*. The media to final grind relationship should be in the 40-fold range, which is a 4-fold particle size reduction in the media mill, multiplied by a 10:1 media to initial particle size ratio.

Examples for choosing media size include:

6.4 microns (7.5 Hegman) * 40 fold = 256 microns or 0.25-mm beads.

13 microns (7 Hegman) * 40 fold = 520 microns or 0.5-mm beads.

25 microns (6.5 Hegman) * 40 fold = 1,000 microns or 1-mm beads.

2. *Divide the media diameter by* 10. Use the optimum 10: 1 ratio of media to initial particle size to determine the size of the product that is to be premilled.

Examples of selecting a premill grind requirements include:

0.25 mm (250 microns) \ 10 = 25 microns (6 Hegman). This is achievable in a rotor/stator mill.

0.5mm (500 microns) \ 10 = 50 microns (4 Hegman). This can be done in a high-speed disperser, but a rotor/stator mill will do it much faster.

1mm (1,000 microns) \ 10 = 100 microns (0 Hegman). This should be achievable in a high-speed disperser.

3. *Other examples*. To achieve a 7.5 Hegman, use a rotor/stator mill capable of a 6-Hegman grind, followed by one pass media mill with a 0.25-mm media. A 0.5-mm media could be used to achieve the desired grind, but it probably would require a finer premilling or an increased residence time. It is recommended to test these two different sized media along with the capabilities of the rotor/stator mill.

To obtain a 7 Hegman, use a rotor/stator mill or a high-speed disperser capable of a 4-Hegman grind, followed by a one-pass media mill with 0.5-mm media.

To achieve a 6.5 Hegman, use a rotor/stator mill or a high-speed disperser capable of a 0 Hegman, followed by a one-pass media mill with 1-mm media.

Typical Premilling Concepts

Here are some practical examples of rotor/stator uses in the coatings industry.

Flexographic Ink

Goal: 0.001% grit on 325 mesh screen (off scale NPIRI).

Problem: Production had problems breaking down beaded black pigment, which caused them to use three passes in the media mill after premixing in a blade mixer. The first pass through helped break the beads, while the second helped to displace some of the air.

Solution: The rotor/stator premilling step was able to burst the beaded black and deaerate at the same time. It took one hour in the rotor/stator mill to eliminate two passes in the media mill, which reduced processing by 14 hours.

Organic Yellow for PVC

Goal: Reduce pigment loading while maintaining color strength.

Problem: The customer was using a blade mixer, followed by a media mill without problems, but he wanted to see if he could improve dispersion and develop a higher color strength.

Solution: The only change made was to replace the blade mixer with a 9,000-foot-per-minute rotor tip speed (8,500 rpm with a 4-inch diameter rotor) rotor/stator mill. A 15% increase was achieved in color strength at the same throughput. The customer was then able to cut the pigment loading by that same 15%.

50% Phthalo Blue Mill Base for Automotive Paint

Goal: 7.5 Hegman and one pass on a media mill.

Problem: Manufacturing had to use a blade mixer for one hour, followed by a 2- to 2.5-mm ceramic horizontal for eight hours in the media mill, followed by a 1.3- to 1.6-mm media mill for nine hours. The total processing time was 17 hours.

Solution: The 1,000-cps viscosity was too low for the blade mill to be effective. It is common that the premix step is not even checked to see what kind of grind is achieved. After analyzing that they were not even on scale on the blade mixer, the customers knew they needed about a 6-Hegman premix.

They also theorized that if they could get a 6-Hegman feed to the media mill, they could use a 0.25-mm micro-media mill. If the premix is not fine enough, the larger particles will clog the micro-media mill. The rotor/stator mill was able to obtain a 6.5 Hegman and eliminate the intermediate media mill. The processing time was reduced to seven hours and 45 minutes, thanks to eliminating one pass and the faster rate through the micro-media mill.

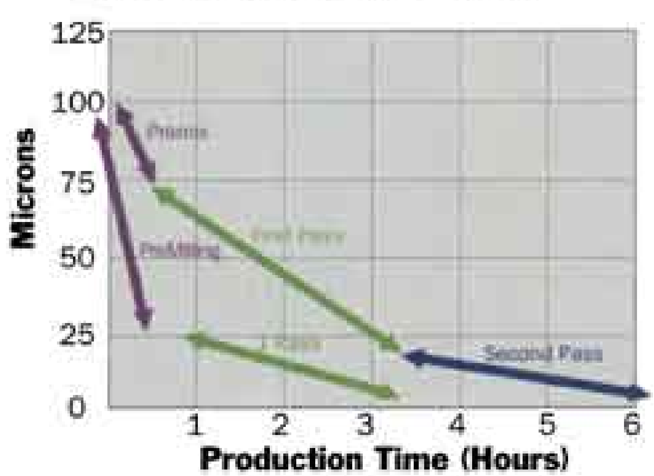
Red Oxide Mill Base for Coatings

Goal: 7.5 Hegman.

Problem: The premix would settle out and clog the two-pass media mill. Solution: The rotor/stator mill was able to get a 5.5 Hegman and suspend the pigment, so that it would not clog a 0.45-mm micro-media mill. The combination of a rotor/stator mill with the micro-media mill eliminated one media mill pass.

Fig 4 **Production Time Versus Final Particle Size**

Comparison of one versus two media mill passes



There are a number of factors that affect dispersions. There is no singular piece of equipment capable of efficiently processing all cases. The design engineer must be knowledgeable of all systems to recommend the best equipment for each process. Do not view the premix as a way to wet out pigments anymore. MacKay and Rubin reported that "surprisingly, some companies use low-speed mixers to do premix preparation for milling."

Solution: In this situation, the subsequent media mill must first complete the predispersion or wetting of the solids before it can even begin to accomplish what it was intended to do.

Media mills can only be as good as what they are fed. The advantages of using rotor/stator mills for premilling are as follows.

- Higher production rates.
- Lower cost per gallon of product.
- Easier cleaning due to the elimination of multiple media mills.
- Allows the use of smaller, more efficient media to increase capacity and/or achieve a finer grind.
- Eliminates settling in the premix.
- Deaerates the premix to allow more efficient final milling.
- Eliminates clogging of small media mills.
- Can increase color strength and reduce pigment loading.

Fig. 5 **Milling Processes Comparison**
(Beaded black dispersing to 7.5 Hegman (6.4 microns) - 100 gallons)

	System 1	System 2	System 3
Premix Stage			
Premix Device Premix (hp)	Blade (20 hp)	Blade (20 hp)	Rotor/Stator (50 hp)
Premix Time	0.5 hr	0.5 hr	0.75 hr
Premix Size Hegman (microns)	0 (100)	0 (100)	6 (25)
Premilling Stage			
Premilling Device	none	Media Mill	Rotor/Stator
Premilling (hp)	—	(30 hp)	(50 hp)
Media Diameter	none	1 mm	none
Premill Size Hegman (microns)	0 (100)	6 (25)	6 (25)
Media/Particle Size		10/1	No Media
Premill Time	–	0.1 hr	included in premix
Media Milling Stage			
Media Mill Passes	2 passes	1 pass	1 pass
Milling (hp)	30 hp	30 hp	30 hp
Media Diameter	1 mm	0.25 mm	0.25 mm
Media/Particle Size	10/1 50+/1 (2nd pass)	10/1	10/1
Milling Time	6 hrs	3 hrs	3 hrs
Total Time	6.5 hrs	3.6 hrs	3.75 hrs
System Price (approximate)	$55,000	$100,000	$70,000
Cleanup Time	Medium	High	Low
Total Horsepower	50hp*2nd pass	80 hp	80 hp

David Ulrich is Midwest regional sales manager for Kady International. He has a bachelor's degree in mechanical engineering and has nine years of experience with media mills and rotor/stator dispersers. James A. Schak is Northeast regional sales manager. He has a bachelor's degree in chemical engineering and is a member of AIChE. He has published a number of technical papers on mixing, drying and reactions and has 19 years of experience with chemical processing equipment. Bob Kritzer is vice president and has a degree in industrial engineering. He has more than 30 years of experience in dispersion with rotor/stator technology. Kritzer has also published numerous technical papers on dispersion equipment.

The KADY PreMilling Process

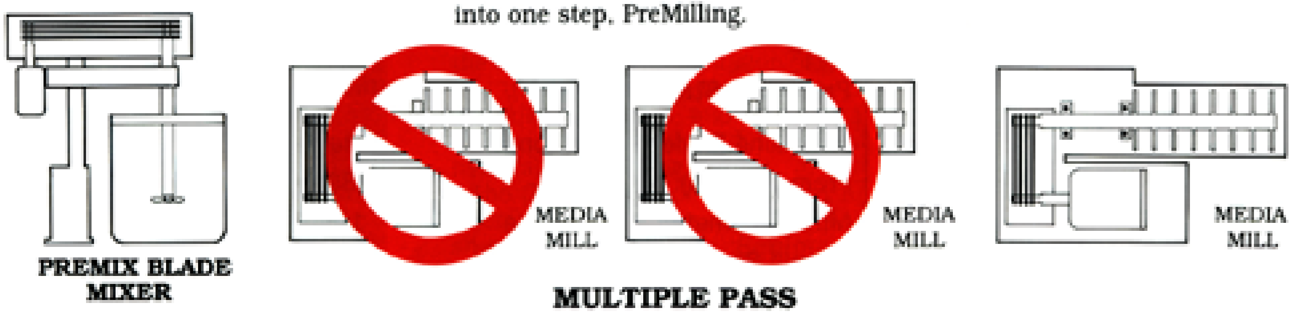
ELIMINATE MULTIPLE MEDIA MILL PASSES

KADY PreMilling, a new processing concept from KADY INTERNATIONAL, combines premixing and early milling stages into one efficient step. Kady PreMilling quickly delivers a finer premilled base to your media mill thereby:

- **Eliminating multiple media mill passes**
- **Increasing Capacity with a higher flow rate**
- **Reducing production time and equipment costs**
- **Saving clean up time and costs**

Typical Premix Media Mill Process:

Coating manufacturers have responded to requirements for finer grinds with the addition of multiple passes, alternative media types, or special multi-chamber high flow media mills. While the media mill remains the best choice for the "final grind", the process becomes inefficient when, typically, the media mill is expected to take particles of 100 microns or more and reduce them to a final size of less than a micron. A more efficient process can be achieved by combining the "premix" and early milling stages into one step, PreMilling.



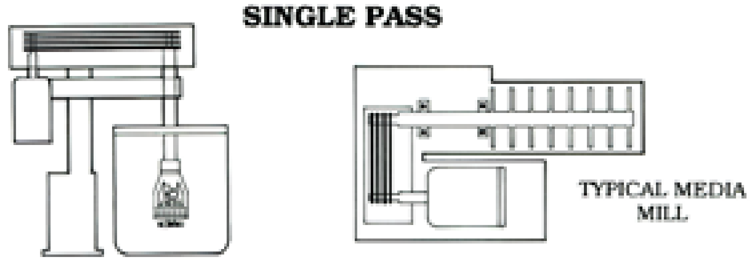
MULTIPLE PASS

The Kady Premilling Process:

KADY PreMilling quickly and efficiently delivers a premilled base to the media mill for the "final grind" - a single high speed KADY Mill can provide a homogeneous "premix" and initial milling below 20 microns in less time than a typical pre-mix; and the time required to finish the grind in the media mill will be greatly minimized. Multiple media mill passes can be eliminated thereby reducing production and clean up time as well as duplication of expensive equipment. The KADY Mill employs a precision, close clearance rotor-stator turning at 9,000 FPM. This tremendous energy input into the batch, (80% faster than other rotor-stators) leads to the superior dispersion results.

SINGLE PASS

PreMilling Process



Call today for more information from the dispersion experts at KADY INTERNATIONAL on new developments such as KADY Sweep Arms, or multi-stage continuous mills that further enhance the advantages offered by KADY PreMilling. Call for details on "in plant" testing, rentals or custom mixing solutions.

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It's All In Our Head.

The KADY Dispersion Head: High-Speed Dispersion for High Quality Color.

The KADY rotor-stator dispersion head can cut production time by up to 90%. In fact, with rotor tip speeds of 9,000 feet per minute, KADY's high-impact technology is a dramactic improvement over blades or props. You get a superior end-product in a fraction of the time -- at a fraction of the cost.

KADY PreMilling saves 30 to 50% in production time.

KADY PreMilling combines premixing and early milling stages into one efficient step. KADY PreMilling delivers a fine, premilled base to the media mill thereby eliminating multiple media mill passes and greatly reducing production time, cleanup and equipment costs.

Take the KADY Challenge

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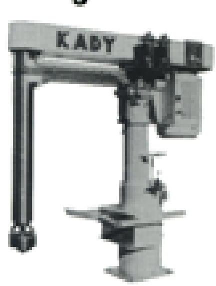

The 25T may be used with a variety of portable tanks. Continuous or batch operation.

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