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MODERN PAINT AND COATINGS, A



Techniques for Efficient Pigment Dispersion Operations

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To achieve better pigment dispersion, first learn to dentify the problems, and then select the proper milling equipment to carry out the solution. $\mathbf{P}_{\text{IGMENT}}$ manufacturers provide a product that has an optimum particle size for its purpose, whether we speak of a 20-micrometer whiting or a hundredth-micrometer black. Because of inter-particle attraction, the dry pigment consists of comparatively large clusters of primary particles.

The purpose of the pigment dispersion operation is the separation of the clusters (known as agglomerates) into a stable suspension of primary particles having the desired physical and optical properties.

It is this apparently simple operation that accounts for a large proportion of coatings manufacturing costs. The paint manufacturer is, or should be, interested in utilizing his dispersion equipment at its highest efficiency consistent with the necessary properties of the finished coating. Since the dispersion operation is concerned with pigment, high efficiency implies high pigment through-put.

"Dispersion" is a generic term, as used by the coatings manufacturer, which does not fully describe the state of the material. A dispersion may be deflocculated, flocculated or agglomerated — or a combination of the three.

How does the pigment happen to as-

sume one or a combination of these three states? The most efficient paint mill made cannot yield the desired dispersion unless the correct conditions exist in the pigment-binderthinner mixture. The paint mill only supplies energy in a suitable form. The desired dispersion will not be achieved unless the proper proportions of pigment, binder and thinner are specified for a given piece of milling equipment.

For the purpose of this discussion, we will emphasize the gross physical aspects of dispersion, since many day to day manufacturing problems are usually not solved with "magic ingredient X" but by simply considering an engineering approach. Although research is revealing more about the chemistry and physical chemistry of dispersion (wetting), much of this aspect of the problem is still Edisonian in approach and not really amenable to a systematic, generalized discussion.

Pigment Agglomerates

As an aid to understanding the dispersion operation, let us consider how a model pigment will behave when we attempt to disperse it in a liquid.

As we mentioned previously, a pigment "as received" will be an agglomeration of loosely held primary particles of the correct particle size. Let us assume that our model pigment consists of uniform spheres of a single size. We may represent this condition by the diagram of Figure 1.



Figure 1 — Pigment agglomerates.

In its simplest form, we may consider the agglomerate to be a mass of material containing thousands of interstices. Our objective in the dispersion operation is to effect the penetration of a liquid into the pores so that the surface of each primary particle is capable of being wet by the liquid. At the same time, enough energy must be applied to the agglomerate to separate and disperse the primary particles in the liquid.

Let us examine one of the agglomerates a little more closely in an effort to logically define some parameters involved in wetting and dispersion. Consider the pores in the agglomerate to be minute capillary tubes, as shown in Figure 2.



Figure 2 — Capillary in an agglomerate.

Poiseuille's Law describes the flow of a liquid through a capillary:

$$\frac{V}{t} = \frac{\pi p R^4}{8 \eta L}$$

V = volume R = radius of capillary t = time η = viscosity p = pressure L = length of capillary

If we imagine the interstices in a mass of pigment to be a maze of capillaries, then the Poiseuille equation confirms what the experienced formulator already knows — that rapid initial wetting is obtained with a low viscosity liquid. This action is further modified by the physical characteristics of the pigment, in this case capillary radius and length.

The larger the pigment particle size, the larger is the capillary radius, "R," the faster the liquid will penetrate the agglomerate. A highly agglomerated pigment will have a longer capillary length, "L," leading to slower penetration of the liquid. These physical concepts have led to the modern, easily dispersed pigment.

An important physical property of a pigment that must be considered for efficient dispersion is its oil absorption.

Oil Absorption

The oil absorption of a pigment refers to the weight of liquid taken up by a given weight of dry pigment to form a paste or coherent mass. There are two general ways of measuring this property. The "rub out method" involves mixing the pigment with increments of oil under very intensive mixing until the point is reached where the pigment is just held together in a stiff mass. This method is exemplified by ASTM D-281.

The second method also involves mixing pigment and oil, but the end point is described as being a soft paste. ASTM D-1483 is descriptive of this method.

Both tests are subjective to some extent, and reproducible results will depend upon a skilled and experienced operator. Oil absorption is expressed as "pounds of oil per 100 pounds of pigment."

The two methods will rank pigments in the same order. The rub-out method yields the lower results by 20 to 40 per cent, depending upon the pigment. Our discussion will be based on the rub-out method, since our experience has been confined to its use and interpretation.

The oil absorption test is a measure of the void volume of a pigment and the amount of oil *adsorbed* on the pigment surface. The void volume usually accounts for the greater proportion of the oil absorption. Another way of describing oil absorption would be in terms of the amount of liquid a pigment will immobilize. Depicted graphically with our model pigment, the oil absorption end point would appear as in Figure 3.



Figure 3 - Oil absorption end point.

The void volume of a pigment or mixture of pigments is a very important property that can markedly effect the technical and economic properties of a coating. Since oil absorption is a measure of void volume, a more meaningful way of expressing this property would be in terms of volume relationships. In other words, it is more meaningful to know the volume of liquid absorbed by a given volume of pigment than to know the weight relationship.

As an example, a titanium pigment might have an oil absorption of 20 pounds of linseed oil per 100 pounds of pigment. Therefore:

The Pigment Volume Concentration (PVC) of this pigment at its oil absorption end point would be:

$$PVC = \frac{2.93}{(2.93 + 2.58)} 100 = 53.2\%$$

To distinguish this PVC as a particular property of the pigment the term Critical Pigment Volume Concentration (CPVC) is used. CPVC, then, is the measure of the void volume plus adsorbed oil of a pigment or a mixture of pigments. The concept of a CPVC was first suggested in 1949 by Asbeck, Laiderman and Van Loo and further refined and expanded by F. B. Stieg.

The CPVC is a reflection of the pigment(s) size and size distribution. In Figure 4 are shown CPVC curves for mixtures of titanium dioxide and extenders. Since the extenders are of a larger particle size than that of the titanium dioxide, packing occurs in which the finer titanium dioxide particles fill the void spaces between the larger extender particles until a very dense condition is reached, creating a maximum CPVC for each extender-titanium dioxide mixture.

There have been many practical applications of the CPVC concept in explaining coating properties; for example, hiding power, washability, du rability, ink receptivity, corrosion resistance and viscosity. Our concern here will be with its effect on the viscosity of the pigment-vehicle mixture, and, in turn, the effect of viscosity on dispersion.

It is generally recognized that the viscosity of a suspension of particles in a liquid will increase as their concen-





ration increases. Although Einstein is pest known for his work on the Theory of Relativity, he posed a relationship between viscosity and particle concentration in 1911 that is still valid:

 $N_r = 1 + 2.5c$ $N_r = relative viscosity =$ suspension viscosity liquid viscosity = volume fraction of particles in suspension

This relationship deals with a relatively small concentration of uniform spheres, a condition that is seldom encountered in practice. The pigments we attempt to disperse are of varying size and size distribution.

We are really interested not only in how the concentration of the pigments will affect viscosity, but also in how their size and size distribution will affect viscosity. To put it another way, we want to know how much of the liquid (in which we are dispersing pigment) is immobilized by this apparently complicated interplay of concentration, size and size distribution.

Some work done in our laboratory indicates that it is possible to determine the parameters that influence the viscosity of a suspension containing a neterogeneous mixture of pigments.

This relationship is based upon dispersions of a variety of titanium dioxideextender mixtures in liquids of different viscosities (see Figure 5).

Here, relative kinematic viscosity is related to the ratio of pigment volume concentration and the difference between pigment volume and CPVC; the denominator could be interpreted as the amount of liquid immobilized by the pigment. It is important to realize that this relationship is based upon pigment-vehicle mixtures that had Newtonian flow.

This relationship may be used as a basis for formulating a pigmentvehicle-thinner composition for a particular mill. For example, the kinematic viscosity of the mill base for a particular mill could be established from the experience with that particular piece of equipment. It only remains to establish two other parameters, the kinematic viscosity of the vehicle in which the pigment(s) is to be dispersed and the CPVC of the pigment or pigment mixture. These parameters can be determined rather easily with a viscosity measurement for the vehicle and an oil absorption measurement for the pigment. By inserting these values in the formula: 510 Ca

$$\log V_r = .027 + \frac{.510 \text{ Cp}}{\text{Pc} - \text{Cp}}$$

the optimum pigment loading (Cp) can be calculated.

Milling Equipment and Dispersion

An original description of the action of milling equipment in breaking up agglomerates has been given by Mr. Temple Patton. He classifies milling equipment as "smashers," "smearers" or hybrids of the two. His classification is given in Table I.

This classification is based primarily on the pigment technologist's viewpoint and may not reflect actual operation of the equipment. Nevertheless, we are concerned with discussing the most efficient means of dispersing pigment and if the action of the mills is considered in relation to the viscosity, this is a valid classification.

Let us examine some typical mills and their operation in a little more detail.

Three-Roll Mill

The three-roll mill is the true "smearer" since the pre-mixed dispersion is fed through a set of rolls operating at differential speeds rotating in opposite directions with a very small clearance between the rolls. Tremendous shearing action is exerted on the pigment agglomerate, causing it to break up into primary particles.

Both tackiness of the feed material and viscosity are important considerations for this type of mill and the penetration of the liquid into the pigment agglomerate is of little or no importance. Therefore, the liquid portion will be composed of all or a high percentage of resin (or binder) solids having a viscosity of 25 to 100 poise. Pigment loading will be as high as possible commensurate with the ability of the mill to handle very viscous materials.

A study of a variety of mill base compositions for three-roll mills shows a rather consistent pattern of pigment loading. For low absorption pigments (such as titanium dioxide, chrome yellows, iron oxides and some extenders) a rule of thumb calculation of 60 per cent of the CPVC would be a starting point for the volume loading of pigment. The liquid portion, as mentioned previously, should be of high viscosity.

The three-roll mill has certain advantages and disadvantages, which are summarized in Table 11.

The high speed impeller mills are exemplified by machinery manufactured by Cowles, Hockmeyer and Day. They consist of a specially designed disk that rotates in a suitable tank at a peripheral speed of 4,000 to 5,000 feet per minute. The disk may be saw-tooth with the teeth alternately bent at 90° to its radius. Other designs are based on several disks with suitable openings through which the mixture is circulated.

The action of this mill has been classified as a "hybrid" by Patton. In a properly designed mill, operated correctly and containing a suitable formulated mill base, both smearing and impact are believed to take place. Smearing will occur in the area between the rotating disk and the bottom of the container; it could also take place in those mills that use a disk of parallel plates where the mill base is forced between the plates. To a lesser extent, smashing will occur by pigment agglomerates impinging on one another during the high rate of circulation in the tank — a sort of "selfgrinding" action.

The magnitude of both smearing and smashing is comparatively low compared to three-roll mills and Kady mills. The high speed impeller mills are high speed mixers and, as such, place more demands on the pigment and the proper design of the grind base.

Our earlier remarks on the effect of liquid viscosity in effecting penetration of the pigment agglomerate are very pertinent to this type of mill. The viscosity of the liquid portion of the mill base should be in the order of one to four poise — a heavier viscosity will only result in "drowning" the pigment agglomerate in the liquid and the energy of the mill will be consumed in simply turning over the viscous liquid.

Again, we need to know what effect the pigment loading, particle size and particle size distribution will have. Based on our experience with titanium dioxide and extenders, and based on results reported in the literature on other pigments, we have found that a rule of thumb for optimum dispersion would be:

Pigment Loading (Volume) =

0.75 CPVC The objective in dispersing pigment

Viscosity	Smash	Hybrid	Smear
Very high			Three-roll mills
			Banbury (sigma) mills
			High speed stone mills
Medium		High speed im	
		Sand mills	
		Ball and pebble mills	
low	Kady mills		

Advantages	Disadvantages
Handles very viscous materiols (i.e., 100% solids coatings). High quolity dispersions. Disperses difficult pigments (i.e., organic colors).	Three-stage operation (i.e., pre-mix, mi and let down to finished product). Moderate to low pigment through-put.

on this type of mill is to achieve laminar flow in the mixing tank. This provides maximum transmission of energy to the mill base as opposed to turbulent flow, which results in wasted energy and excessive heat. In order to achieve this end, certain design parameters are usually related to the disk diameter, "D," as shown in Figure 6.



Figure 6 — Geometry of high speed mixers.

In a tank with this design, with the impeller rotating at 4,000 to 5,000 feet per minute (peripheral speed) with a properly formulated mill base, the mill base should attain maximum laminar circulation under maximum power load, resulting in a "doughnut shaped" pattern with the center portion of the blade visible.

As in every case, this mill has certain advantages and disadvantages, as enumerated in Table 111. The high speed impeller mills have very definitely made an impact on the coatings industry.

Ball and Pebble Mills

Ball and pebble mills comprise one of the oldest types of dispersing equipment. Offhand, one might think that they would be classified as smashers because of the importance attached to cascading and impact of the balls on the pigment agglomerate. In reality, however, both smashing and smearing take place and a properly formulated mill base will balance these two forces.

Too much smashing will result in excessive wear of balls and liner, while too much smear will result in inefficient dispersion.

Proper loading of the mill with balls and charge is necessary. Experience shows that the mill should be charged to contain 30 per cent ball volume, 20 per cent mill base volume and 50 per cent vapor space.

In the case of "conventionai" ball nilling, the liquid portion of the grind ase would have a viscosity of about one to two poise. The low viscosity will lead to rapid penetration of the pigment agglomerate. Pigment loading can be related to CPVC again by the following relationship: Pigment Loading (Volume) =

0.55 CPVC Advantages and disadvantages of the ball mill are shown in Table IV.

Sand mills are logical extensions of the ball mills in the sense that they are highly efficient ball mills. It is well known that the efficiency of ball mills can be increased by reducing the size of the balls, since more impact faces will be available. A limit is reached, however, where the balls will be immobilized and no dispersing action will take place.

This limitation has been overcome in the case of the sand mill by pumping the mill base through a moving bed of very fine balls (sand). The moving sand exerts a shearing action on the mill base with the probability of some smashing by sand and pigment.

The sand mill requires a pre-mixing operation to which some attention should be paid.

The force exerted on a pigment agglomerate is roughly proportional to the ratio of the sand diameter to the agglomerate diameter. It follows, then, that agglomerates larger than the sand particles (20 to 40 mesh) will tend to resist being dispersed to primary size. In other words, a relatively good quality pre-mix is needed to be fed into the mill. In practice, this is often accomplished by making up the pre-mix on a Cowles dissolver.

The ratio of sand to mill base is important. A ratio of one to one is desirable; higher ratios result in dilatant mill bases, which may freeze the mill in extreme cases. A lower ratio will reduce the efficiency of the mill.

The formulation of the mill base is very similar to the formulation for the ball mill. In fact, a properly formulated ball mill base can work very well in the sand grinder. The liquid portion would be one to two poise. Although the same pigment loading can be used, a slightly lower pigment loading is often preferred:

Pigment Loading (Volume) =

0.50 CPVC

Advantages and disadvantages of the sand mill may be summarized as shown in Table V.

The Kady mill is the true smasher of

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Table III — Advantages and Disadvantages of High Speed Impeller Mills	
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Advantages	Disadvantages
One-step dispersion is possible (i.e., no pre-mix or let-down).	Difficult ta disperse ultrafine pigments (i.e., arganic and black).
Very high through-put, (i.e., pigment dispersion is complete in 20 minutes).	Difficult to attain superior dispersion with- out careful selection of raw materials and skilled operator.
Relatively law capital investment.	For best dispersion often requires finer,
No parts to replace except the inexpensive impeller.	better classified pigments, which may be more expensive.

Table IV — Advantages and Disadvantages of Ball and Pebble Mills

Advantages	Disadvantages
No pre-mixing required. Volatile loss is eliminated. Minimum skill to operate. Can handle all types of pigments and achieve quality dispersions.	Through-put is camparatively low. Charge volume fixed by mill size. Occasional prablems in emptying and let-down.

Table V — Advantages and Disadvantages of Sand Mill

Advantages	Disadvantages
Can handle all types of pigments. Very high quality of pigment dispersion. Fairly high through-put.	Requires reasonably good pre-mix. Difficult to clean and change colors.

Table VI — Advantages and Disadvantages of Kady Mill

Advantages	Disadvantages
Rapid wetting and dispersion.	Let-down can be risky unless care is exercised.
Easily cleaned with solvent wash.	Through-put is rather low.

pigment agglomerates. Consequently, very low viscosity is desired so that resistance of the mill base in passing through the slots of the head is reduced. In theory, the pigment could be dispersed in the volatile portion of the grinding liquid, but this could lead to subsequent problems in let-down because of a tendency to pigment flocculation. In practice, it is normal to disperse the pigment in a low nonvolatile liquid (Ca ten per cent solids) followed by additional non-volatile added in the mill to stabilize the dispersion before let-down.

The liquid portion for the Kady mill will be about one poise. Pigment loading is not critical, but should not be excessive nor so low that throughput suffers. Probably, the relationship is:

Pigment Loading (Volume) =

0.40 CPVC

Some advantages and disadvantages are listed in Table VI.

Throughout this discussion on dispersion we have talked about volume relationships. This approach seems most valid to us, since mill throughput is a volume relationship and most coatings are sold by volume and are applied by volume.

The CPVC relationship is a more meaningful value than oil absorption since it tends to place the proper emphasis on the pigment volume rather than on its weight. For example, titanium dioxide having a density of 4.2 and an oil absorption of 18 would have a CPVC of 55 per cent. An iron oxide with a density of 3.0 and an oil absorption of 26 would have a CPVC of 54 per cent. A phthalo green with a density of 1.9 and an oil absorption of 37 would have a critical pigment volume concentration of 57 per cent.

As far as dispersing equipment is concerned, our discussion would indicate that all of these pigments should have nearly the same loading by vol-

Table VII — Behavior of Deflocculated and Flocculated Dispersions

Property	Deflocculated	Flocculated
Flow	Newtonian — long and stringy.	Thixotropic, or with a "yield value" — short ond puffy.
Opticol properties	Fully developed — moximum hiding, tinting strength.	Degroded — tinting strength weak, but may be increased by mild agitation. White pigments tend to be yellow tone.
Settling	Slow, but to a compact layer, difficult to redisperse.	Fairly fast, but to loosely com- pacted layer, easily redispersed.
Flooding	Likely if pigment is fine particle size, law density, law viscosity.	May occur, but will not be cured until flocculation is cured.

ume. This is not apparent if only the oil absorption is considered.

Evaluation of a Dispersion

The dispersion operation, then, is directed to reducing the pigment agglomerates to their primary particle size so that full development of the properties of the pigment may be realized. The means of determining whether this goal has been reached often leave someting to be desired.

For example, in its crudest form, the evaluation of a dispersion can involve spreading some of the freshly milled grind base with a spatula over a flat surface. A more refined method is the Hegman gauge, in which the grind base or coating is spread in a wedgeshaped slot and precise-sounding numbers are used to indicate the size of the "specs" or agglomerates visible to the eye.

If we consider titanium dioxide that has a primary particle size of 0.2μ , and if we consider a 7 Hegman grind (0.0005 inch) as being satisfactory, we can still have particles of 0.0005 inch or larger buried in the deeper end of the gauge. These particles would be ten μ or larger — that is, 50 times larger than the optimum size for this pigment.

Taking this a step further, consider a phthalocyanine blue of a primary size of 0.05μ . At a 7 Hegman grind, those agglomerates that are visible to the eye are 200 times the size of the primary particle of the blue pigment.

The question, then, is what are we seeing when we evaluate a dispersion by a fineness of grind gauge. Fortunately, the picutre is not as dark as it seems.

In the case of our titanium dioxide ground to a 7 Hegman, we are seeing less than 0.01 per cent of all the particles in the coating that fills the slot. In the case of the blue pigment, we are probably *seeing* about the same *per*- *centage*, but this tells us nothing about the particles we don't see. Putting it another way, a 7 grind may be entirely satisfactory for titanium dioxide, but entirely unsatisfactory for phthalocyanine blue.

The quality of a dispersion is probably best measured by what we don't see. This brings us back to the beginning of our discussion, namely, that a dispersion is best evaluated in terms of the function a pigment is to perform — that is, tinting strength, tint tone, gloss, hiding power.

It is well worth the effort to determine through intensive milling exactly what a pigment is capable of and then to use this level of hiding power, tinting strength or texture (in the case of extender pigments) to evaluate the efficiency of dispersion under production conditions. A target is needed even though under practical conditions economy or equipment or time may be against achieving this target.

Condition of a Dispersion

Assuming that we have handled our pigment correctly on the type of milling equipment we have available, and by one measure or another have determined that the dispersion is satisfactory, we still must make a finished, salable product by the addition of more binder, thinner, driers, flow-control agents and suspension aids. Unfortunately, this procedure often destroys the properties of the pigment that have been carefully developed through proper dispersion techniques.

A common problem is that of pigment flocculation, in which we have at some stage dispersed the pigment to its primary size but for one reason or another the primary particles tend to loosely clump together. How can such a condition be recognized? Based on our own experience and the experiences of others, we can list certain behavior typical of deflocculated and flocculated dispersion (see Table VII).

These are some of the outward, indirect indications of the conditions of a finished dispersion. What can be done to ensure a dispersion that will be trouble-free, economical and salable?

Dispersion to Finished Product

The let-down of the finished dispersion is one of the first places we can look for trouble. Some of the causes of flocculation we have found are:

(1) Water and other immiscible polar compounds, introduced deliberately or inadvertently through contamination.

(2) Immiscibility of vehicles, solvents or both, which leads to agglutination where the pigment is trapped in one phase. Compatibility of all liquids should be determined before attempting to pigment them.

(3) Proprietary agents, including flow control, suspension and wetting aids. The flow control and suspension aids often act simply because they flocculate the pigment. As for wetting agents, they are very useful and also very specific. It is well worth the extra effort to determine exactly how much is required; too much or too little can flocculate the pigment.

(4) Pigment "shock" or seeding after let-down can often be traced to the relative solubilities and concentrations of solvent and binder in the grind base and let-down liquid. Because of a concentration gradient between grind base and let-down there is a tendency for solvent to migrate from the grind base to the let-down. This is particularly true of the more modern type of milling equipment in which a larger concentration of solvent is present in the grind base. Compare a three-roll grind with a sand mill grind. This condition is further aggravated with strong solvents. The solution to this problem is to disperse in the weakest possible solvent or to mix grind base and let-down immediately, preferably under strong agitation.

Aqueous Dispersions

Our discussion so far has been pointed in the direction of pigmen dispersion in organic media. With regard to aqueous dispersions, same general principles apply.

Our conditions for fast wetting of pigment agglomerate can be applicaliterally to water, which has a low v cosity and a high surface tension Why, then, is it so difficult to obtain satisfactory dispersion in water? The answer lies in the electrostatic force

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which increase tremendously as the dielectric of the medium increases. Stabilization is achieved by adding an electrical charge to the surface of the pigment in the form of a dispersant.

As was mentioned previously, the amount of dispersant required is very critical; too much as well as too little can cause pigment flocculation. We can study this very easily for titanium dioxide by determining its dispersant demand.

This method simply involves the preparation of a pigment-water mixture (usually at 70 per cent pigment) and then titrating this mixture with small amounts of dispersant. After each addition of dispersant, the viscosity is measured (on a Brookfield Viscometer, for example). A plot of dispersant addition against viscosity will give us curves similar to those in Figure 7, showing a minimum viscosity point. The dispersant concentration at this point is called the dispersant demand of the pigment and is the level of dispersant required to prevent flocculation of the pigment in water.

We also stressed the importance of oil absorption in dispersion. A similar situation exists in aqueous dispersions — this is known as water demand and is defined as the amount of water required to fill the pigment voids. In our laboratory it has been related to oil absorption by the following relationship:

Water Demand =

16.7 + 0.67 (O.A.) The addition of the correct amount



Figure 7 — Dispersant demand.

of dispersant will lower the water demand by about ten per cent.

Summary

We have discussed a basic property of pigments, CPVC, and its relation to the proper formulation of grind bases. Pigment dispersion was discussed in terms of a model pigment in an attempt to assist the reader in visualizing some ideas on dispersion.

A classification of milling equipment was discussed from the point of view of the action of the mill as a "smasher," "smearer" or a hybrid. Note that in true smashers or smearers the pigment concentration is of secondary importance, primary consideration must be given to the vehicle viscosity — low and tack-free for smashers, high and tacky for smearers. The hybrids place more demands on the pigment quality and loading — the vehicle viscosity is of the same order of magnitude for the hybrids.

Dispersion problems were discussed from the point of view of recognizing the problem. No specific solutions were offered since, in our experience, no broad solution is possible — each case is specific and must be treated specifically.

Aqueous dispersions are basically similar to organic dispersions when the dispersant demand of the pigment is understood.

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