INTRODUCTION

Working With Iron Oxide Pigment In Water Suspension

During the past 18 years, iron oxides water suspensions and slurries have been gaining significant market acceptance in the process of adding color to concrete mixers, especially for the production of concrete masonry. Iron oxide suspensions are versatile in automation and present cleanest operating conditions. The following is a presentation of the theory, technology and requirements for using a wet system.

Coloring With Iron Oxide Pigments

Synthetic (manufactured) iron oxide pigments are in the form of particles ranging approximately from 1/10th of a micron to 1.0 micron. The difference in color between one pigment and another is due to the shape and surface structure of the particle. The characteristics of the surface of each particle determines which of the color components in the light that is striking it will be reflected and thus recognized by the eye. This is the reason why pigments appear different in color when subjected to different light conditions, i.e., daylight vs. artificial or northern vs. southern skylight.

Light from these various sources have different components (wavelengths) which when selectively reflected by the same pigment will not appear to the eye as the same color.
Iron oxides, when used as a colorant, work by masking the surface they are covering so that the eye will see the particles of the pigment or more specifically recognize the light component being reflected by it, thus establishing a particular shade.

It is important to recognize that the particles of dry pigment do not exist individually. Electrostatic forces and packing compaction cause the individual particles to clump together to form agglomerates ranging in size from 20 to 150 microns with the majority falling between 60 and 120 microns.

**Mass Tone versus Undertone**

When a surface is totally covered with particles of a certain pigment, so that the eye can only see the pigment, the color is then at its maximum strength. Adding more pigment would only cover other pigment particles and is wasted. At this point the color is at its saturation point, which is labeled **MASS TONE**.

When the amount of pigment used to cover an object is not enough to “mask” the total surface, the eye in this case will be seeing both the color of the pigment as well as that of the uncovered portion of the surface. If the original surface is neutral in color (white or grey), then the pigment becomes diluted and appears weak with a shift in tone. The new shade is referred to as the **UNDERTONE** of the color. On the other hand if the original surface is colored, then the eye will perceive the mixture of the two as a third color.

Pigments of the same family, such as reds for example, could have different **UNDERTONES**. Upon dilution with a white extender, reds may appear purple or pink. The reason for this is that a red pigment particle, depending on its structure, may reflect the red component of light plus a certain amount of blue (purple undertone) or some yellow (pink undertone). The appearance of the undertone effect depends on the reflected amount of these secondary components relative to the mass tone when diluted. In this case the **MASS TONE** gradually loses its predominance in favor of the **UNDERTONE**.

The undertone characteristics of a color also determine if the mass tone, such as red, would have a yellow (terra cotta) or blue (deep red) appearance.

Yellow oxides have, in general green or red undertones. Black oxides could have red or blue undertone.

**Mixed Colors**

The generally used synthetic iron oxide pigments, in concrete application, are produced in three basic colors: **RED - YELLOW - BLACK**
However, each of the above three is available with a family of undertones, which in turn are different from one manufacturer to another.

When two or more pigments are mixed, a new color is developed. The following are basic combinations used by the concrete product producers.

- **BUFF**................................. **YELLOW (PREDOMINANT) + RED**
- **ORANGE**.............................. **RED (PREDOMINANT) + YELLOW**
- **LIGHT BROWN**...................... **RED + YELLOW + BLACK (EQUAL PARTS)**
- **DARK BROWN**....................... **RED + YELLOW + BLACK (OVER 50%)**

**Color Accuracy**

In general, the accuracy of a commercial iron oxide pigment is controlled to within +/- 5% in strength and undertone. These limits can be tolerable, although not desirable, when a single color is used. However, it becomes far more critical and less acceptable when a blend of different pigments is used.

For example, assume a color blend of yellow, red and back made of equal parts of the three (light brown). If the components accuracy is +/- 5% then the following 2 combinations could occur:

**Combination #1**  
**YELLOW** +5  
**RED** -5  
**BLACK** -1  

**Combination #2**  
**YELLOW** +1  
**RED** -1  
**BLACK** +5  

While the above two colors are expected to be the same, color #1 will be rich in yellow, weak in red, with a 10% spread from the standard between these two components. Color #2 will be rich in black while the yellow and red components are close to standard. Color #2 will appear darker than the standard.

It is obvious that a much closer tolerance of the pigments is desirable to produce products with consistent colors. An accuracy of +/- 1.0% will limit the color spread to 2%, a variation not easily detected by the eye. This is particularly necessary in a process where many other factors are present to affect the final color.

**Coloring of Cement**

When concrete is colored, only the cement component takes the color. The color of the other components simply act as diluents in the mixture. Because the particle size of iron oxides are of the same magnitude as that of cement, the color saturation is reached quickly and, depending on the quality of pigment used, 8 to 12 parts color to 100 parts of cement by weight will result in maximum coloration in most cases. The practical limit, however, is 6 to 8 percent.
Dilution of color by the cement occurs when less than the above level is used and, as discussed before, the undertone of the pigment will become prevalent. Thus a red color will appear purple if it has blue undertone and is added to the cement in small quantities.

**Effect of the Grey Cement on the Final Color**

High color loading in concrete means that all, or a high percent, of the cement particles are masked. Therefore variation in the greyness of the cement has less pronounced effect on the final color.

Low color loading in concrete, however, will favor the undertone of the pigment used. Because not all of the cement particles are covered, the eye will mix the color of the pigment and that of the cement. Technically the grey color of the cement can be equated to a weak black pigment which when added to the color of the pigment used, will produce a new blend. It follows that if the color of the cement changes, then the resulting color of the final blend will be different. Therefore, the consistency of the color of the cement becomes more crucial with the decrease of the pigment loading.

Light color-shades are affected in different ways than darker colors by the greyness of cement. A dark brown has a high percentage of black in the blend, and is affected less than light browns where the present amount of black is much lower. Therefore dark shade colors are influenced much less than lighter shade colors by the change in the greyness of the cement.

The color of the aggregates, sand and stones, have its own effects although to much lesser extent. The human eye is an excellent mixer of color. When we look over a block of colored concrete, such as a paver, we do see a mixture of the color of all the particulates present. Since the size of the stones is relatively large when compared to the sand or cement particles, it is usually covered by the other components and the overall color of the block is not affected. It is to be noted that this is only true when a concrete block is not split and we are only looking at the molded surface. The much smaller size of the sand particle, however, does influence the final color but in different ways. If it is dark in color it can act as a week colorant. The amount of sand, its size and the presence of its fines develop, in combination with the cement, different macro structures that would stretch or compact the amount of pigment used and therefore influence the color. High color loadings will mask this effect and so would the lack of fines in the sand. In order to achieve color consistency, the grey shade of the cement must be closely controlled and to a lesser degree the uniformity of the color and consistency of the sand.

**Factors Affecting Color Development Concrete**

The coloring strength of a pigment is set by the manufacturing process. It is essentially inversely proportional to the particle size. Finer particles have higher tinting strength. Once the pigment is manufactured, its tinting strength and color is established and cannot be altered. This does not mean that all iron oxide pigments of the same color have the same strength. Actually it depends
on the manufacturing process and quality of the pigment produced. Natural iron oxide pigments, although they share the same chemical form, have larger particles and contain high amount of impurities. Thus natural pigments have lower tinting strength than synthetics.

Iron oxides behave differently under different processing conditions. To this end it is the nature of the host system that determines the final coloring efficiency and results.

One characteristic that affects the behavior of iron oxides is the agglomeration of its particles. As mentioned before agglomeration is the clumping together of several particles due to electrostatic forces between them. Mechanical bridging also results in agglomeration when the pigment is compacted under its own weight, such as when stored on pallets. This condition is readily observed when opening a bag of dry oxide. Clumps or balls of different sizes can be seen mixed with fine powder. The fine powder may also be a mixture of much smaller agglomerates containing fewer particles.

When dry iron oxide pigment is mixed with the medium to be colored, the mixing action will break up the agglomerates into single particles and smaller clumps. The residual agglomerates are the result of

INEFFICIENT MIXING
SHORT MIXING TIME
WRONG PH LEVEL
CHANGE IN OIL ABSORPTION
FLOCCULATION

The effects of inefficient mixing and short mixing time are quite obvious and do not need further discussion.

The PH of a wet system controls the wetting rate of the color particles. Iron Oxide pigments must be thoroughly wetted out in order to develop full color potential. This wetting action is accomplished by the fluid carrier in the mix. Surface coatings contain organic solvents, water or heat liquefied polymers. Concrete mixes contain water for the hydration of cement.

Pigments with low PH level require longer wetting time. In this case difficulty may arise in controlling the development of color in “home made” slurries. However, because a concrete mix is alkaline, this condition would not present a problem when using dry iron oxide in cement coloring. Manufactured pigment suspensions must have adjusted PH levels to perform consistently in this respect.

The oil absorption quality of a pigment is a measure of the amount of water required for full wetting. The difference in water demand due to a change in oil absorption could be significant enough to affect the final color when dry pigment is used in a concrete mix. Difficulty in preparing a high-loaded slurry will also occur if a high oil absorption pigment is used.

Flocculation occurs in dry pigment when introduced into a wet system, where the fluid carrier,
such as water, has less attraction to the pigment particles than the particles have between themselves, thus promoting clumping or secondary agglomeration. It is a similar action to that of oil in water where the oil stays together in large patches.

Regardless of the origin of the agglomerations, the net result is a reduction in tinting strength. As discussed earlier, when particles are stacked on top of each other, the pigment is wasted and becomes inefficient or **UNDERDEVELOPED**.

When mixed long enough and the right conditions are present, all the agglomerates will eventually break down to a minor size and the pigment will attain maximum tinting strength.

The above results can be observed when mixing dry pigment in concrete. Longer mixing cycles develop the color of the final product to a higher intensity, proportional to the mix time. Since the required mixing time for full-color development in a low efficiency system, such as a concrete mixer, is quite long compared to the actual concrete mix cycle, the condition of full color development is rarely achieved. Concrete mixers will develop normally between 70% and 90% of the color strength when dry pigment is used.

**Efficiency of Color Systems**

The efficiency of a color system, whether it is dry pigment, manufactured suspension or slurry on site (home made) depends on how well the pigment has been wetted out and deagglomerated. These two factors determine how effectively the pigment is used.

Pigment in water suspension is not necessarily more efficient than dry. Unbroken agglomeration of pigment if present, in a wet medium would reduce its coloring intensity.

The presence of cement, water and pigment in a concrete mixer can, and indeed most of the time does, encourage balling. Balling is an agglomeration of cement and pigment on a larger scale than that of the pigment alone. However, since balling is readily recognizable, the cycle of the mix is usually adjusted to prevent it from occurring. Balling occurs if water is introduced into the mix before the cement and/or dry color have been well dispersed.

An efficient pigment suspension must contain a balanced system of suspending agents to prevent undue settling, and a dispersant to act against agglomeration, keeping the particles separated at all times for full color development.

The manufacturing process of iron oxide suspension must include maximum separation of the particles through proper mechanical means. Full coating of the particles with the dispersing agent is essential to sustain this condition. A good suspension should have a very high and consistent deagglomeration rate to insure repeatability of the color from batch to batch. An efficient suspension can only approach the optimum color intensity of the pigment used, it cannot exceed the original strength value.
Conversely, dry pigment, or a slurry with a high rate of agglomeration, can be totally dispersed if subjected to sufficiently long mixing time. Eventually full color intensity is achieved and will reach that of an efficient suspension. However, this condition will require a mix time too long for an economical operation. In the case of concrete mixing, the chemical reaction of the mix is compromised if the cement is subjected to over mixing. A pre-manufactured iron oxide suspension is totally prewetted and requires a mixing time well within the economical and mechanical mixing cycle of concrete.

**Mixing Cycle for Colored Concrete**

The water absorption characteristic of the cement turns it into a hydrophilic element that would react with water whenever it is available. When cement, water and iron oxide pigment are present together, water is preferentially absorbed by the cement leaving the pigment in a semi dry stage. This state encourage the clumping together of the pigment particles forming dry balls. This condition of “BALLING” will not breakup totally during the short mixing cycle of the concrete. Large balling creates defects in the concrete product but light balling, although imperceptible to the eye, do decrease the effectiveness of the pigment. Balling can be easily avoided by using the proper cycle for introducing the pigment into the concrete mix.

The underlining principle of a good concrete mixing cycle is the individual spreading of the interacting components before the next component is added. When pigment is introduced into the mix, regardless of where in the cycle it is added, it should be allowed to mix uniformly with the present components before the next step begins. Once spread uniformly, the particles will not clump or reagglomerate, being forced apart by the other components in the mixer. It is of interest to note here that the cement may also clump together if it is not uniformly spread before water is added.

Based on the above principle, the following cycle emerges as the most efficient way to achieve desirable and optimum results.

**Preferred cycle:**

* First the aggregate (stone and sand) are introduced into the mixer and given a short mix
* Next the dry pigment is introduced and mixed. This step should be as long as needed to uniformly spread the color and insure that there are no clumps present. The stone and sand act as grinding medium and further help breakup the agglomerates. When a pigment suspension is used, prewet water may be added to help spread the color.
* Once the color is properly spread, the cement may be added and mixed. Again insure that it is thoroughly spread before going to the next step
* Water is now introduced, adjusted to the proper level and mixed.

The above cycle is particularly important when water suspension of iron oxide is used. If the pigment is not thoroughly spread, the cement will absorb the water from the color suspension, causing it to flocculate and loose efficiency.
Automation

While the sequence of the steps in the cycle is very important, it is also imperative that the timing in adding each component be accurately repeated. Timing insures that no over mixing or under mixing do occur. Automatic color dispensers are recommended here to insure the uniformity and repeatability of adding the pigment to the concrete mix.

Dispensers for Iron Oxide Water Suspensions

The following are four systems that represent the design principle of most of the dispensers used for delivering iron oxide water suspensions to the concrete mixers.

**Dual Probe Volumetric System: Fig. A** This system uses a precision tube with two probes, one is fixed in low position and the second is adjustable. The liquid color is pumped into the tube until it reaches the adjustable high probe, which acts as a shutoff switch to end the fill. Upon demand, the liquid is pumped out of the tube until it reaches the lower fixed probe which in turn ends the dispensing. The volume set between the two probes is the amount being metered. This design does not require emptying the delivery hoses which introduces inaccuracy of the measurement. One tube is required for each color component used.

**Single Probe Self-flush Volumetric System: Fig. B** This system uses a precision tube with one fixed water probe and one adjustable liquid probe. Water is first fed into the tube until it reaches the lower fixed probe, which provides a base line for the color. Liquid color is then added until the level reaches the adjustable high probe where it will stop. Upon demand, a valve in the bottom of the tube is opened and the diluted color is gravity fed into the concrete mixer. A water shower is next started to clear and clean the tube from any liquid left on the wall. One tube per color is required.

**Volumetric Single Tube with Electronic Probe: Fig. C** This system requires one tube to measure all the color components. The probe is a sonic unit that delivers an analog signal of the color height to a computer that controls the various levels of the components. Water is first introduced to form a base line for the first color. The first color is introduced and its level is determined by the formulation programmed in the computer. The second and third color (if used) follow in the same manner. Upon demand a valve is opened in the bottom of the tube, gravity feeding the content into the mixer. A shower follows to clear and clean the tube.

**Weigh Cell Single Tube With Computer Control: Fig. D** A single tube is used to measure all the color components. The tube is mounted on a weigh cell to measure and control, through a computer, the components being measured. The colors are fed in sequence according to a formulation in the computer. Upon demand, air pressure is used to move the color, via a hose, to the mixer. Water under pressure is next used to flush the remaining color in the tube and hose.