Process Design and Optimization of External Additive Blending on to Toner Surface

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Abstract

Additive blending is one of the most important steps in the toner manufacturing process. As toner sizes get smaller and the toner shapes and surfaces get more controlled, the step of blending plays a much greater role in imparting flow and charging functionality to the toner. For a given toner and additive formulation, the blending process significantly impacts the charge level, charge rate and the powder flow which are critical to xerographic performance. A functional analysis of the blending process is of considerable use in order to better understand the critical parameters involved and how they are related to functional performance of resultant powder in xerographic machine. A methodology for functional analysis and identification of critical parameter is presented for achieving good blend quality. The good blend quality is determined by toner/additives mixture harmony, uniform dispersion and distribution of additives and the optimal attachment of additives. Methods for the characterization of blend quality are presented. The additive attachment functionality is represented by the strength of the attachment on to the toner surface. The "Weak" symbolizes under-attachment (i.e., loosely attached or free additives), "Medium" represents functional level of attachment (optimal attachment strength) while "Strong" represents over-attachment i.e., additives are buried into toner surface and are non-functional for flow. This methodology takes into account the kinetics of additive attachment, dynamics of blend process and the heat transfer involved. Based on these understanding, we have proposed a set of process critical parameters that takes into account the kinetics, dynamics and the heat transfer. This methodology is demonstrated to be successful for process scale-up. The paper also talks about various proprietary blend tools and the dynamics imparted by these tools. To understand the dynamics of the blending process, it is important to quantify the forces acting on the batch to be blended. The blenders commonly used for toner additive blending are fluidizing mixers where the high rotational speed of the mixing tools fluidizes the batch of material. This allows all of the particles, regardless of particle size, density, coefficient of friction, and other characteristics, to intermix and disperse very quickly as low viscosity liquids. Hence it is plausible to consider aerated powder in a blender as a pseudo homogeneous phase. Thus, the theory applied to describe the dynamics of liquid mixing can be applied to solids blending by treating the air-solid mixture as a pseudo-homogeneous phase in a high speed mixer. To apply the liquid mixing theory to solids blending, we must be able to define the pseudo-homogeneous physical properties of aerated powder in the blender, and ignore, for the moment, the consequences of compressibility and inertially-driven stratification. A single phase computational fluid dynamic (CFD) model is presented for understanding the flow pattern generated by different tools. Several metrics have been proposed to evaluate these tools by performing parametric design using CFD.

A. Introduction/Background

The definition of toner-additive blending is to create a *harmonious mixture* of toner and additives where additives are *uniformly dispersed/distributed* over and *attached* to the toner surface.

The above toner-additive blending definition has three key features which must be satisfied for functionality of toner-additive blend:

a) Harmony of mixture – implies additive and toner working well together. This is a task of toner designers to ensure compatibility between toner and additives for desired functionality - hence proper selection of additive for a toner formulation would provide "mixture harmony".

b) Uniform dispersion/distribution of additives on toner surface – implies breaking of additive agglomerates to primary aggregate size and distributing it uniformly particle-to-particle over the toner surface – metrics: "mixture variance" - characterized by macroscopic additive composition measurement in several samples pulled randomly from the mixture and "additive surface coverage" - characterized by Scanning Electron Microscopy.

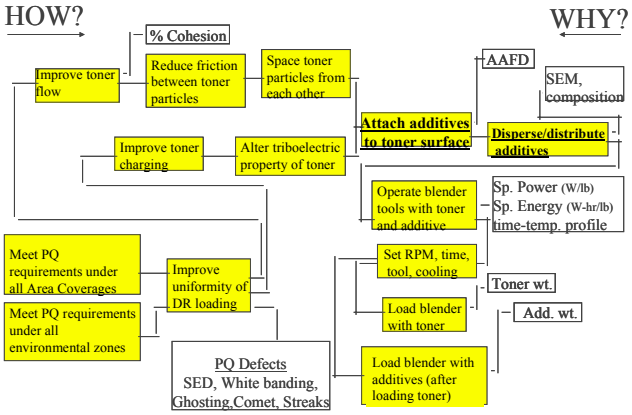
c) Additive attachment to toner surface – implies strength of additive adhesion to toner surface – metric: "Additive Adehesion Force Distribution (AAFD)" technique as described in US patent, where strength of additive adhesion is characterized by amount of additives *removed* at low sonication energy ("Weak" adhesion) and amount of additives *retained* at high sonication energy ("Strong" adhesion). The level of sonication energy is chosen depending on toner-additive formulation and must be relatable to functional performance of toner in xerographic processes.

A "good blend" must have all these three features optimized for a particular xerographic system.

Functional Analysis of Blending Process

A functional analysis of the blending process would be of considerable use in order to better understand the critical parameters involved in blending process and how they are related to functional performance of resultant powder in xerographic machine. A functional analysis can be constructed using the Functional Analysis System Technique, as described elsewhere. A so-called FAST Diagram of the additive blending process for a mono-component toner is shown in Figure 1. The flow of functions is shown by yellow colored boxes while the metrics associated with each functions are shown in white colored boxes. As we follow the flow of functions from left to right in the diagram we keep asking the questions "How we do it?" and the flow from right

to left asks? "why we do it?" Once the flow of functions is identified we associate one or more metric to each of these functions.



These metrics are the potential critical parameters for the above system. Critical parameters for the blending process are the measurable variables in the process (control factors) which directly affect the functional performance of the resultant powders. Critical parameters for the resultant powders are the measurable functional properties that directly affect the xerographic performance of the powder in the xerographic hardware. The potential critical parameters for the above-mentioned system are listed below:

Blend process

1. Toner and additive types and amounts
2. Specific Power
3. Specific Energy
4. Temperature-time profile

Blended Powders

1. SEM
2. Additive macro-composition
3. Additive Adhesion Force Distribution (AAFD)
4. Cohesion (%)
5. Print Quality Defects Metric – Side Edge Deletion, White Banding, Comets, Streaks etc.

The next step would be to narrow down this list to few critical parameters. This is enabled by creating an IOC (Input Output Constraint) chart, cause and effect diagram (Ishiwaka Diagram) and performing designed experiment using the potential critical parameters. Based on succesful designed experiments and improved understanding of the system the potential critical paremeters list is narrowed down to selected few "critical parameters". These parameters must be tracked and maintained during the development of blend process in order to ensure successful scale-up of process to manufacturing. In this paper we would focus on the evolution of "critical parameters" for the blending process. For a fixed toner/additive formulation these parameters are:

1. **Specific power (Watt / lb)** – defined as "power drawn / batch weight" i.e.,

   Specific Power = (Load Power – No Load Power) / (Batch Weight) ….(1A)

   This parameter captures the "**dynamics**" of the blender at any given time i.e., the forces acting on the batch at any given instant.

2. **Specific energy (Watt-hr / lb)** – defined as

   Specific Energy = $\int$ (Specific Power) x (Blend Time) …. (1B)

This is the amount of energy imparted to the blend batch on per unit weight basis. For constant specific power, this is equivalent to "(Specific Power) X (Blend Time)". Since blending is a transient process, this parameter captures the"**kinetics of the process**" once the "dynamics" has been defined.

3. Batch temperature-time profile (°F vs. minute) – Change in batch temperature with time as a result of energy dissipation in the batch. This parameter captures the "**thermal characteristic**" of the process.

Blending as a Transient Process

a) Additive Attachment Kinetics

Additive blending is a batch process and the blend time is dependent upon the additive attachment kinetics. The additive attachment kinetics can be described as:

$$\text{Weak} \xrightarrow{K_1} \text{Medium} \xrightarrow{K_2} \text{Strong} \qquad \ldots.(1C)$$

Where "Weak", "Medium", and "Strong" symbolize strength of additive attachment as they evolve over time in the blending process. The kinetic constants K_1 and K_2 control the kinetics of the attachment and are dependent on toner-additive formulation as well as the blending process intensity (i.e., the rate at which work is done on toner surface). The "Weak" symbolizes under-attachment (i.e., loosely attached or free additives), "Medium" represents functional level of attachment (optimal attachment strength) while "Strong" represents over-attachment i.e., additives are buried into toner surface and are non-functional for flow. Based on this description it is apparent that functional level of additive attachment ("Medium") should be maximized. By this technique, the weakly adhered additives termed "Weak" and strongly adhered additives termed "Strong" are directly measured and reported in percent (%). Hence, Medium = 100 – (Weak + Strong). During the blend process development, one should optimize the "Medium" for optimum functional performance of powder.

Assuming that the kinetic is first order, the rate of change of Weak (W), Medium (M), and Strong (S) can be mathematically represented as:

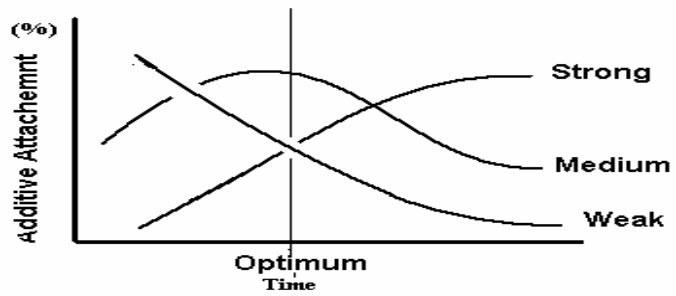
$$(-dW/dt) = K_1 (W) \qquad \ldots.(1D)$$

$$(dM/dt) = K_1 (W) - K_2 (M) \qquad \ldots.(1E)$$

$$(dS/dt) = K_2 (M) \qquad \ldots.(1F)$$

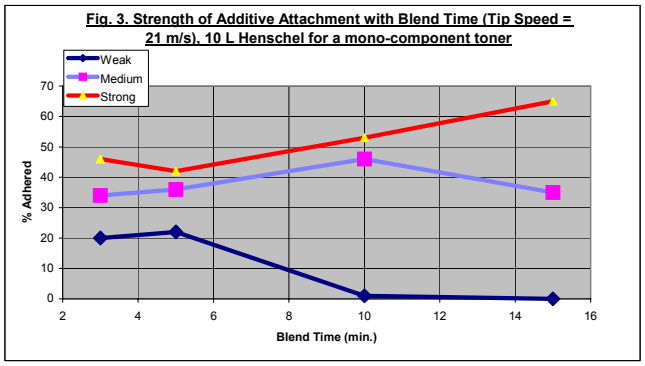
$$\text{Also, } M = 100 - (W+S) \qquad \ldots.(1G)$$

Based on these expression, the W, M, and S would show a trend with time as shown in the Figure 2.



According to this kinetics, the "W" will exponentially decrease with time while "S" will exponentially increase with time. The Medium "M" will show a maximum at certain time. This may be considered the optimum blend time.

In the following figure (Figure 3), the "Weak", "Medium" and "Strong" is tracked for one of the mono-component toner blending. The figure shows that at a given blend tool RPM (fixed K_1 and K_2), as the blend time increases the "Weak" goes down, "Strong" goes up while the "Medium" shows a maximum around 10 minutes blend time. Hence, 10 minute blend time at the present RPM is considered optimum for additive attachment. At the optimum blend time, the "weak" has also diminished to an acceptable level i.e., the level of loose/free additive is minimal. This trend of additive attachment is consistent with first order kinetics as described above. It was also noticed by SEM that at 10 minute blend time, the additive dispersion and coverage had reached an optimum.



b) Thermal Transients

In the blending process, mechanical energy is applied to a closed system which manifests itself in the rise of batch temperature due to friction among particles. Excessive batch temperature could cause formation of coarse particles and leads to over attachment of additives due to increased additive impaction at higher toner surface temperature. For mono-component toners made of polyester, the experiences suggest that the batch temperature should be kept below 105°F. If the critical temperature is not known, one must maintain the batch temperature below the blocking temperature of toner. Further, one should prepare several blends with "Hot" jacket temperature at Pilot Scale in order to push the batch temperature to the limit of "critical batch temperature" and identify potential failure modes for toner functionality.

Heat Transfer Model

To account for the rise in batch temperature with time at a given operating condition, a blender heat transfer model is proposed. The heat balance over the entire blending system at any instant can be represented as:

(Rate of energy input by blend tool) = (Rate of energy accounted for heating the batch) + (Rate of energy accounted for heating the cooling medium) + (Rate of energy accounted for heating the blender wall) + (Rate of energy lost to the ambient)

This expression indicates that only a fraction of total energy input by blend tool accounts for the temperature rise in the batch. A simple heat balance is proposed here which accounts for the "rate of energy input to the batch" causing the measured rise in batch temperature:

$$dT_b/dt = q'/(\bullet\rho_{mix}*Cp_{mix}) - h*As*(T_b-T_j)/(\rho_{mix}*V*Cp_{mix}) \quad (2)$$

Rate of change Heat generation rate Heat transfer rate

dT_b/dt : differential of the batch temperature with respect to time (K/sec)

h: Blender wall to batch heat transfer coefficient (W/K*m^2)

As: heat transfer area (cooling jacket area) (m^2)

T_b: Batch Temperature (K)

Tj: Jacket Temperature (K)

ρ_{mix}: Density of the pseudo-homogenous phase {powder + air} (Kg/m^3)

ρ_{mix}= volume fraction of toner (ε_s) *$\bullet\rho_s$ + volume fraction of air ($\boldsymbol{\varepsilon}_{air}$) * ρ_{air}

V: Volume of the blender (m^3)

Cp_{mix}: Specific heat of the pseudo-homogenous phase {powder + air} (J/Kg K)

Cp_{mix}= mass fraction of toner * Cp_{toner} + mass fraction of air * Cp_{air}

q': Heat generation per unit volume which accounts for heating the batch (W/m^3) = (Total heat generated due to dissipation of power) – (Energy required to heat the jacket cooling water) – (Heat leakage to ambient)

$\boldsymbol{\varepsilon}_s$: Volume fraction of toner = M / (V $\boldsymbol{\rho}_{toner}$)

M: Batch weight (Kg)

$\boldsymbol{\rho}_{toner}$: Specific gravity of toner (Kg/m^3)

ρ_{toner}(TCD) = 1200 Kg/m^3, ρ_{toner} (SCD) = 1700 Kg/m^3, Cp_{toner} = 2200 J/Kg K , Cp_{air} = 1006.4 J/Kg K

In formulating the above heat balance, the following assumptions have been made:

1. The cooling jacket temperature reaches a steady-state in a very short time compared to the blend process time i.e., over the processing time the jacket inlet and outlet temperature remains constant.
2. The wall conduction resistance to heat transfer is negligible (this assumption is valid only when the Biot number is <<1).
3. The batch temperature of the blender is constant with respect to axial and radial directions at any given time. A high degree of

turbulent mixing in the blender ensures uniformity in the batch temperature along the radial and axial directions.

4. The specific heat generation in the batch which accounts for batch heating (q') and heat transfer coefficient between bed and wall (h) is constant over time.

This model was validated by performing experiments and the results are shown. All 9 experiments were run for 30 minutes in order to let the batch reach a steady-state temperature. A two components toner (9.5 micron) along with silica additives (3% by wt. equivalent to 100% theoretical SAC) were used for these experiments. The batch temperature was recorded at 0, 0.5, 1, 2, and successive 2 minute intervals for 30 minutes. Figure 4 shows the batch temperature vs. time for varying jacket temperature and RPM at a fixed loading. One can see that all curves show a rise in batch temperature (transient) which ultimately levels off (steady-state) with respect to time. The transient part of the curve is used to obtain 'h'. Once 'h' (heat transfer coefficient) is known, the q' (heat generation/volume) can be obtained from the steady-state portion of the curve. As seen in the figure, the model adequately predicts the batch temperature rise with time. The slight deviation at high RPM can be attributed to material build-up (insulating barrier) on the wall which suggests that "h" may not be constant with time contrary to our assumption. The model also showed that the excessive rise in batch temperature can be minimized by reducing solids loading.

Design Rules for Additive Blending

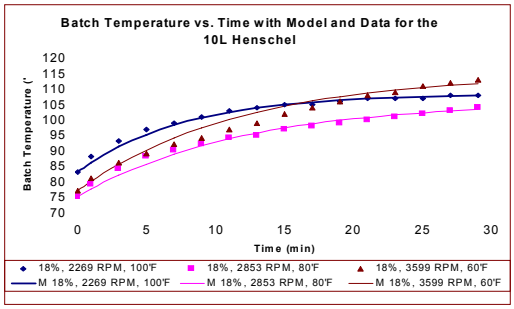
1. ***Equivalent specific energy*** *at or above* ***critical specific power level*** *would yield* ***similar toner functionality*** The specific power and energy is expressed as:
   Specific Power = (P/M) = $N^3 D^2 f(N_{Re}, N_{Fr}$, *Geometric factors)*
   Specific Energy= $\int$ (P/M) dt = $\int N^3 D^2 f(N_{Re}, N_{Fr}$, *Geometric factors) dt*

   This guideline suggests that by matching the "dynamics of mixing" and "kinetics of attachment", the quality is matched.

2. *Maintain batch temperature below a critical level*: This ensures that adverse thermal effects on blend quality is minimized. For a mono-component toner, we have maintained the batch temperature below 105° F. Above 105° F, the additive adhesion becomes stronger due to softening of toner resin for polyester mono-component toner.

The design rules suggest that the *higher specific power (intensity) blender is more effective because it would achieve desired specific energy level in shorter blend time*. Thus, the blenders which provide higher specific power at a given RPM would also enable shorter blend time thereby increasing productivity.

Critical power is defined as the minimum amount of power required to break the additive agglomerates to primary aggregate size range. The primary aggregate of additives are primary particles joined by chemical bonds and these primary aggregates form a chain type structure (see the attached Fig. 9). However due to short-range inter-particle forces these aggregates combine to form agglomerates. Thus, one can perceive "critical power" as the minimum amount of external force imparted by blades which is required to overcome the interparticle forces between primary additive aggregates and keep them apart. To understand the concept of "critical power", four mono-component toner blends were made at equal specific energy (P t / M) but at different levels of specific power (P/M) on 1200 L Littleford blender. The specific power was changed by changing the tool speed and specific energy was made equal by compensating for blend time. Thus, a smaller specific power blend was blended for a longer time compared to a higher specific power blend to yield equal specific energy. The SEM's for all the 4 blends were examined. Although, the specific energy levels are equal, it is apparent that the blend made at lower P/M has larger number of agglomerates on toner surface with more bare or "additive-lean" regions. While, higher specific power blends have higher additive surface coverage and better dispersed additives (fewer agglomerates). Thus, for this particular product one could say that the specific power must be maintained above P/M = 74 W/lb in order to achieve desired dispersion and distribution of additives. However, as per the design rule 1, once we exceed the critical specific power, the "equal specific energy" blend should yield equivalent quality. Therefore, in terms of additive dispersion and distribution the difference between SEM # 3 (P/M = 168 W/lb) and SEM # 4 (P/M = 368 W/lb) is little compared to the difference between #3 and #1 (P/M = 74 W/lb).



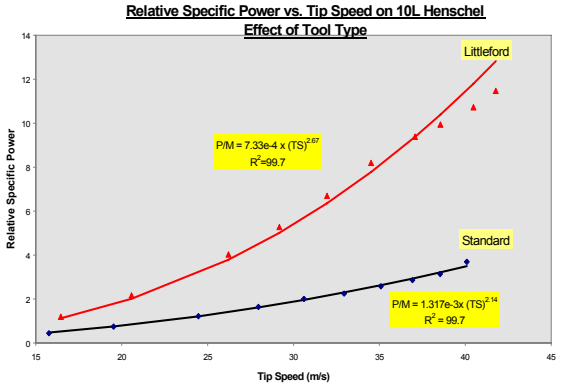
Effect of Tool Design on Specific Power

To accomplish this we recorded power draw while blending a fixed volume loading of toner at various blend tool speeds. In order to obtain the power required to blend the toner/additive system (P), and eliminate the power associated with electrical and mechanical losses, a "no load" power measurement was made at various chosen speeds. This was done prior to placing the toner and additive in the blender. Thus, the power required to blend the toner at a specific tool speed was obtained by

Toner blending power (P) = (Power with toner loading) - (No load power)

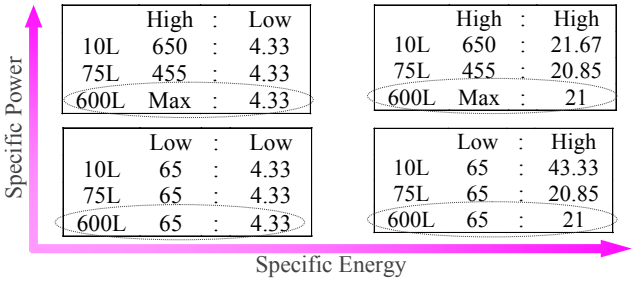
The specific power is obtained by dividing the power draw by batch size. Thus, *the "specific power draw" is a manifestation of rate of work done per unit weight of material. A tool which provides higher power (i.e., rate of work done) is more effective because it would achieve desired specific energy level in reduced cycle time.* Figure shows the difference in performanc between the Henschel "standard" and Littleford "V-tool" configuration on the same vessel. This was enabled by retrofitting the Littleford V-type tool on a Henschel blender. One can notice that Littleford tools are much more intense (higher work rate at a given tip speed) than Henschel tool suggesting that equal energy can be obtained at a much shorter time. Further, the coefficient b=3 for Littleford tool

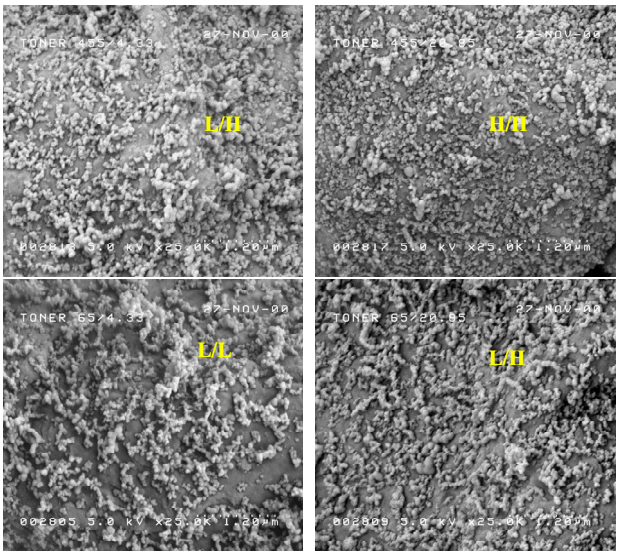
indicates that the mixing regime is “turbulent” while the mixing regime for Henschel tool is “laminar” (b=2).



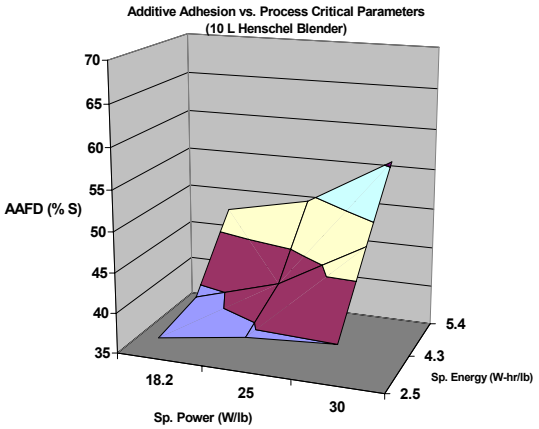
We have also shown that the specific power is independent of solids loading in the blender, thus eliminating the need for using solids loading as a control factor in the optimization of a blender. The solids loading however significantly impacts the batch temperature rise as explained before. Thus, we propose that a blend optimization can be carried out with any blend tool configuration by performing a designed experiment in specific power and specific energy domain while keeping the solids loading at nominal 15% (based on toner specific gravity). This approach has been successfully employed at Xerox for various toner designs.

A relevant example is shown and the mapping of additive adhesion and SEM for this design is also shown for 10 L and 75 L.





As expected, one can notice that strong attachment goes up as the specific power and energy goes up. This was also validated with the scale-up.



Conclusion

A methodology for functional analysis and identification of critical parameter is presented for achieving good blend quality. The good blend quality is determined by toner/additives mixture harmony, uniform dispersion and distribution of additives and the optimal attachment of additives. Methods for the characterization of blend quality are presented. The process optimization methodology has been successfully applied for the scale-up of process.

Author Biography

Samir received his Ph.D. in Chemical Engineering from the Ohio State University. He joined Xerox Supplies Organization in 1993 and is currently an Area Manager responsible for upsteram Toner/Developer Designs and process technology. Samir was the leader for advancing the additive blending process for both conventional and chemical toners at Xerox. He has designed several novel blend tool that are used for manufacturing toners. He has authored 8 external technical papers and over 15 U.S. patents.