

CCA Effects on the Triboelectric Charging Properties of a Two-Component Xerographic Developer

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Abstract

Charge control agents (CCA's) are frequently added to toners to create a desired charge level and polarity. Triboelectric charging tests on model, CCA-surface-treated toners and carriers indicate that CCA transfer between toner particles and carrier beads can be a controlling factor for the magnitude and polarity of toner charge — in extreme cases, toner polarity can even be reversed as a result of CCA transfer. The tests also indicate that the transfer process (and hence the final charging properties of a toner) can be significantly affected by the concentration of the toner in a xerographic developer.

Introduction

The sign and magnitude of toner q/m can be manipulated by the addition of charge control agents to the toner recipe (CCA's — typically salts formed from bulky inorganic or organometallic ions paired with small counter-ions¹). While CCA's are usually melt-mixed into the bulk of the toner resin matrix, they are presumed to affect toner charging performance via their partial presence at the toner surface^{2,3}. Indeed, some CCA effects are best explained in terms of CCA transfer from toner particles to the surface of the carrier beads. For example, for a model negative polarity toner, the sign and magnitude of q/m has been linearly related to the amount of CCA species transferred to the carrier surface⁴. An additional example of CCA/carrier effects can be deduced from charging data taken on a developer based on a positive-charging toner⁵ — for that system, the sign of the toner charge actually reverses at low toner concentrations (i.e., the extent of CCA transfer from the toner particles to the carrier beads is a function of the toner concentration). Similarly, an indirect example of CCA transfer to carrier particles is the enhanced developer conductivity that can result when a conductive carrier is paired with a CCA-containing toner⁶ (an effect where the carrier packing density is enhanced by the lubricating effect of the transferred surface CCA).

To illustrate the importance of CCA/carrier effects on the triboelectric charging performance of CCA-based toners, experimental toner charging data will be presented and analyzed, using model developers based on CCA-containing toners, on matching base CCA-free toners, and on toners and carriers directly treated with CCA's.

Theory

Conceptually, toner triboelectric charging can be simply expressed as a product of terms related to the physics and chemistry of charging and to the mechanics of mixing^{7,8}, e.g.:

$$q/m = (A'/(C + C_0)) \cdot (\phi_{\text{toner}} - \phi_{\text{carrier}}) \cdot (1 - \exp\{-\gamma t\}) \quad (1)$$

where q/m is the toner charge to mass ratio, at a toner weight % concentration of C , and A' and C_0 are constants related to the size and density of the toner and carrier particles. The rate constant γ defines the rate of triboelectric charging, and is related to the mode of toner/carrier mixing process. The ϕ_{toner} and ϕ_{carrier} terms represent the charging tendency of the toner and carrier particles, and the relative magnitude of these two terms directly governs the polarity of the toner particles, and also affects the magnitude of the toner charge.

Since the surface of toner and carrier particles are typically composed of several components (e.g., binder resin, pigment, coating, etc.) the ϕ_{toner} and ϕ_{carrier} terms may be expressed in terms of surface-weighted contributions from the various components⁷⁻¹⁰, e.g.:

$$\phi = \mathbf{P}_i \mu_i + \mathbf{P}_j \mu_j + \dots \quad (2)$$

where the total sum of the fractional weights, \mathbf{P}_i , \mathbf{P}_j etc., equals unity, and the parameters μ_i and μ_j etc. are characteristic charging factors for the various surface components.

Frequently, the mechanical forces of the triboelectric mixing process affects the surface composition of toner and carrier particles — i.e., the \mathbf{P}_i , \mathbf{P}_j values are functions of the mixing time, and the resultant q/m values can be complex non-linear functions of mixing time^{5, 8, 10}.

For a CCA-containing toner, the ϕ_{toner} term can be expressed as:

$$\phi_{toner} = P_{CCA} \cdot \mu_{CCA} + (1 - P_{CCA}) \cdot \mu_{toner}$$

or

$$\phi_{toner} = P_{CCA} \cdot (\mu_{CCA} - \mu_{toner}) + \mu_{toner} \quad (3)$$

where the charging contributions from the toner binder resin, pigment etc., are all combined in a single composite μ_{toner} term. (Equation (3) is not based on any particular charge exchange mechanism — it merely indicates that a CCA affects the tendency of a toner to accept or donate charge).

The effect of a CCA on toner polarity will depend on the relative magnitude of the μ_{CCA} and μ_{toner} terms; e.g., if $\mu_{CCA} > \mu_{toner}$ then Equations (1) and (3) indicate that ϕ_{toner} will increase as the surface content P_{CCA} increases, and that the toner q/m will become increasingly positive. Experimentally, however, levels of CCA above some optimum value can sometimes lead to a decline in q/m^2 , and for a carrier-toner pair that produces a positive polarity toner, this CCA effect may reflect a CCA-driven increase in the value of $\phi_{carrier}$, as given by:

$$\phi_{carrier} = P_{CCA} \cdot \mu_{CCA} + (1 - P_{CCA}) \cdot \mu_{carrier}$$

or,

$$\phi_{carrier} = P_{CCA} \cdot (\mu_{CCA} - \mu_{carrier}) + \mu_{carrier} \quad (4)$$

where, in this case, P_{CCA} is the fraction of the carrier surface covered by CCA that has transferred from the toner particles, and where $\mu_{CCA} > \mu_{carrier} > \mu_{toner}$.

For the case where CCA is partially transferred from the toner to the carrier particles, there will be an attendant decline in ϕ_{toner} , if the P_{CCA} term of the toner is decreased by the transfer process, and this will also contribute to a decrease in q/m .

If a toner-to-carrier CCA transfer process actually reaches an equilibrium state, then the toner q/m value will eventually stabilize, and such behavior has indeed been observed in long-term toner-throughput aging experiments with CCA-based toners^{2, 3, 11}.

In general, P_{CCA} at any mixing time, t , (for both toner and carrier particles) can be described by:

$$P_{CCA,t} = (P_{CCA,0} - P_{CCA,\infty}) \cdot \exp\{-k \cdot t\} + P_{CCA,\infty} \quad (5)$$

where $P_{CCA,0}$ and $P_{CCA,\infty}$ are the values of P before and after extended mixing. As a result, ϕ_{toner} and $\phi_{carrier}$ will vary as:

$$\phi_{toner,t} = (\phi_{toner,0} - \phi_{toner,\infty}) \cdot \exp\{-k_{toner} \cdot t\} + \phi_{toner,\infty} \quad (6)$$

$$\phi_{carrier,t} = (\phi_{carrier,0} - \phi_{carrier,\infty}) \cdot \exp\{-k_{carrier} \cdot t\} + \phi_{carrier,\infty} \quad (7)$$

and these non-linear changes in ϕ_{toner} and $\phi_{carrier}$ will produce non-linearities in q/m as a function of mixing time. (For complex interactions, where changes in ϕ_{toner} and $\phi_{carrier}$ may reflect simultaneous, consecutive and/or reversible processes, a comprehensive kinetic equation will contain many terms^{11,12}; however, for any particular

fixed condition, experimental charging data can often be described by simple expressions based on relationships such as those in equations (6) and (7).

Now, while substitution of the terms in equations (6) and (7) into equation (1) will produce a non-linear equation for q/m as a function of mixing time, experimental q/m data can only be used to deduce values of the difference term ($\phi_{toner} - \phi_{carrier}$), and individual values of ϕ_{toner} and $\phi_{carrier}$ can only be obtained with respect to some arbitrary reference. Despite this critical limitation, an in-depth analysis of q/m data from CCA-based developers can yield informative mechanistic insights, and this approach will be illustrated in the Results and Discussion section of this present report.

Experimental Procedures

A nominal 9 μ black toner was prepared by melt-mixing, jetting and size-classification, and approximately 0.25 wt% of a positive CCA was blended into the toner resin matrix in the melt-mix step. For diagnostic tests the toner was also prepared in a CCA-free state. A surface-CCA-treated toner was also prepared from the latter base toner — 0.05g of the positive CCA was blended with 50g of base toner, with small stainless steel balls being used to facilitate the blending process.

The test carrier was prepared from nominal 130 μ steel carrier beads, powder-coated with an approximately 50/50 PMMA/ fluoropolymer mixture. A CCA-coated version was also prepared — 2000g of the polymer-coated carrier were roll-milled with 0.1g of the positive CCA. Finally, an aged sample of the test carrier was recovered from a developer that had been used for an extended print test.

For q/m generation, developer samples were conditioned at 16°C/20% relative humidity, and were agitated on a paint shaker. At regular time intervals, small samples were removed for q/m evaluation using a total blow-off procedure.

Results and Discussion

Test 1: CCA-free base toner with test carrier

Figure 1 shows q/m data taken at a series of fixed toner concentrations from 0.5 wt% to 5 wt% for the base, CCA-free toner and the test carrier. As can be seen, this developer produces a strongly negative polarity toner.

At long-mixing times, equation (1) can be rearranged to give:

$$(\phi_{toner} - \phi_{carrier}) = (q/m) \cdot (C + C_0) / A' \quad (8)$$

For the test developer, $A' = 70 \mu\text{C.g.eV}$ and $C_0 = 1$, and with ϕ_{toner} set at an arbitrary reference value of 0 eV, then the experimental data can be fitted using the $\phi_{carrier}$ values listed in Table 1. (From these values, by definition, $\phi_{carrier} = 1.15 \text{ eV}$, and $\phi_{toner} = 0 \text{ eV}$).

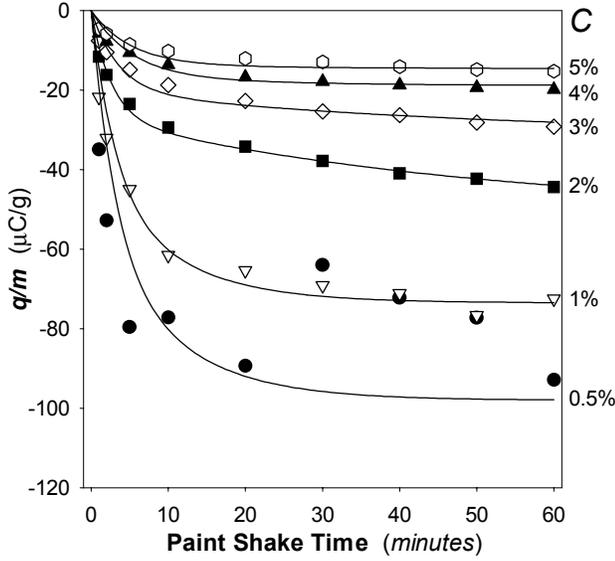


Figure 1. q/m data for the CCA-free base toner with the test carrier. (Note that q/m is negative at all toner concentrations)

Table 1

C wt %	$\phi_{ca,\infty}$ eV	$\phi_{ca,0}$ eV	$\phi_{to,\infty}$ eV	$\phi_{to,0}$ eV	k_{to} min^{-1}	k_{ca} min^{-1}	γ min^{-1}
5	1.25	1.15	0	0	0	0.05	0.15
4	1.35	1.15	0	0	0	0.05	0.2
3	1.80	1.15	0	0	0	0.02	0.3
2	2.20	1.15	0	0	0	0.02	0.4
1	2.10	1.15	0	0	0	0.1	0.4
0.5	2.10	1.15	0	0	0	0.1	0.4

Test 2: Melt-mixed-CCA toner with test carrier

Figure 2 shows the q/m data at high toner concentrations for a developer based on the test carrier and the toner melt-mixed with about 0.25 wt% of a positive CCA. Using the 1.15 eV value for $\phi_{carrier,0}$ (as deduced from Test 1), the data in Figure 1 yield $\phi_{toner,0} = 1.80$ eV for the melt-mixed-CCA-based toner.

However, as shown in the total data set in Figure 3, the developer based on the melt-mixed-CCA-based toner apparently produces a positive toner polarity only at high toner concentrations. In a previous report⁵, we hypothesized that a CCA-transfer carrier enhancement process was necessary to produce a positive toner polarity; however, diagnostic tests made for the present report now suggest that anomalous negative toner polarities seen at low toner concentrations are in fact the result of a carrier poisoning effect, caused by CCA transfer to the carrier surface, and Table 2 lists the results of an analysis based on this hypothesis.

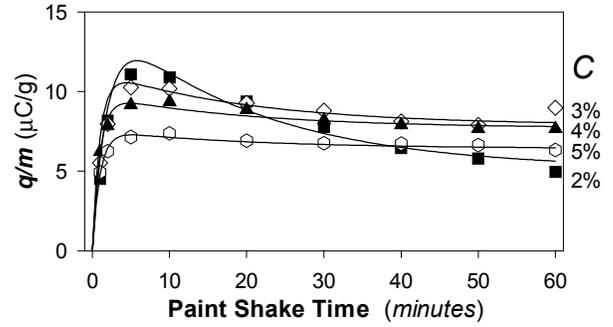


Figure 2. q/m data at high toner concentrations for melt-mixed-CCA toner with test carrier (expanded q/m scale). (Note that q/m is positive at all of the high toner concentrations)

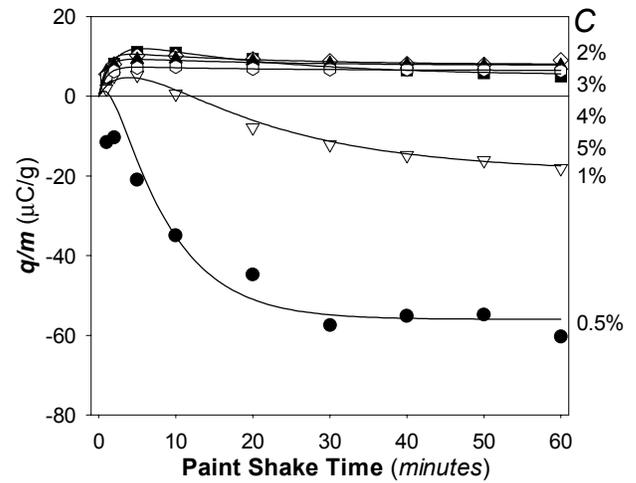


Figure 3. The entire q/m data set for the melt-mixed-CCA toner with the test carrier. (Note the reversal in q/m polarity at low toner concentrations)

Table 2

C wt %	$\phi_{ca,\infty}$ eV	$\phi_{ca,0}$ eV	$\phi_{to,\infty}$ eV	$\phi_{to,0}$ eV	k_{to} min^{-1}	k_{ca} min^{-1}	γ min^{-1}
5	1.15	1.15	1.70	1.80	0.05	0	1
4	1.15	1.15	1.70	1.80	0.05	0	1
3	1.15	1.15	1.60	1.80	0.05	0	1
2	1.15	1.15	1.37	1.80	0.05	0	0.5
1	1.35	1.15	0.80	1.80	0.06	0.5	0.15
0.5	2.00	1.15	0.80	1.80	0.15	0.5	0.15

The analysis indicates that CCA transfer to the carrier is most severe at low toner concentrations, even though such concentrations expose the carrier beads to the lowest total concentration of CCA from the toner particles. One possible explanation is that increased mechanical/frictional forces at low toner concentrations enhance the CCA transfer process. (Significantly, developer aging experiments using CCA-containing toners

have shown a similar effect ¹¹ — developer aging was found to be most severe at a constant low toner concentration).

Given a value for μ_{CCA} , equations (3) and (4) can be used to convert the ϕ values in Table 2 into CCA surface coverage values. From a diagnostic test (discussed in the next section), μ_{CCA} can be deduced to be 4.5 eV, and this value yields a CCA surface coverage of about 40% for the melt-mixed-CCA test toner. In the lowest toner concentration tests, this level falls to about 20%, e.g.:

$$\phi_{toner, \infty} = P_{CCA} \cdot (4.5 - 0) + 0,$$

so for $\phi_{toner, \infty} = 0.8$ eV,

$$P_{CCA} = 0.18.$$

Similarly, the carrier surface becomes 25% contaminated with CCA, e.g.:

$$\phi_{carrier, \infty} = P_{CCA} \cdot (4.5 - 1.15) + 1.15,$$

so for $\phi_{carrier, \infty} = 2.0$ eV,

$$P_{CCA} = 0.25.$$

As a consequence, the increase in $\phi_{carrier}$ to 2.0 eV and the decrease in ϕ_{toner} to 0.80 eV, produce a highly negative toner charge for the predicted q/m value at a toner concentration of 0.5 wt%,

$$(e.g.: 70 \cdot (0.8 - 2.0) / (0.5 + 1) = -56 \mu C/g)$$

Test 3: Surface-CCA-treated base toner

The q/m data in Figure 4 are for the surface-CCA-treated toner with the test carrier. By comparison with the previous results from the CCA-free and melt-mixed-CCA toners, it is clear that a low level of CCA can have a major effect on both toner polarity and charge level, when it is directly applied to the toner surface.

A 1.15 eV value for $\phi_{carrier, 0}$ (as deduced from the previous tests), yields a value of 4.5 eV for $\phi_{toner, 0}$ for the surface-treated toner tests, and the total analysis is given in Table 3. If the initial CCA surface coverage on the toner is assumed to be 100%, then $\phi_{CCA} = 4.5$ eV by definition.

Table 3

C wt %	$\phi_{ca, \infty}$ eV	$\phi_{ca, 0}$ eV	$\phi_{to, \infty}$ eV	$\phi_{to, 0}$ eV	k_{to} min^{-1}	k_{ca} min^{-1}	γ min^{-1}
5	1.15	1.15	4.50	4.50	0.05	0	0.7
4	1.15	1.15	4.50	4.50	0.05	0	0.7
3	1.15	1.15	4.50	4.50	0.05	0	0.7
2	1.45	1.15	4.30	4.50	0.05	0.5	1
1	2.10	1.15	4.10	4.50	0.06	0.5	1
0.5	2.20	1.15	3.60	4.50	0.15	0.5	1

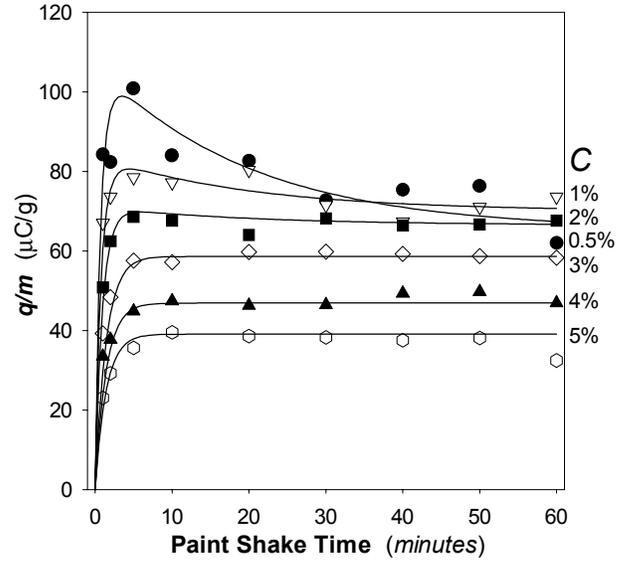


Figure 4. q/m for CCA-treated base toner with the test carrier. (Note that q/m is positive at all toner concentrations).

As in the previous tests, the test data at high toner concentrations show only a minor degree of CCA transfer to the carrier surface, but at the lowest 0.5 wt% toner concentration, the CCA coverage on the carrier is deduced to be about 30%, while the CCA coverage on the toner drops from 100% to 80%.

Test 4: Surface-treated carrier with CCA-free base toner

According to equation (4), a surface-coating of CCA on the carrier surface should increase the value of $\phi_{carrier}$ (up to 4.5 eV for a monolayer coverage), and thereby reduce the q/m level of any toner below that obtained with a CCA-free version of the carrier. For the present CCA-free base toner, where $\phi_{toner} = 0$ eV, a CCA-treated carrier should produce a highly negative toner charge, e.g. at 5 wt% toner concentration:

$$q/m = 70 \cdot (0 - 4.5) / (5 + 1) = -52.5 \mu C/g$$

However, for the test carrier coated with 0.0001g of a positive CCA per gram of carrier, the experimental q/m value is + 35 $\mu C/g$ at a 5 wt% toner concentration of the CCA-free toner, and the developer gives highly positive and stable q/m values at all toner concentrations, as shown in Figure 5.

Apparently, the toner particles rapidly acquire a surface coating of CCA from the CCA-coated carrier beads, and this gain (coupled with a partial CCA-loss from the carrier beads) provides a high level of positive toner charge. The net effect on $\phi_{carrier}$ and ϕ_{toner} is listed in Table 4 (based on an assumption that the carrier is initially 100% coated with CCA), and these values indicate a CCA loss from the carrier of about 90% at high toner concentrations, and about 40% at the 0.5 wt% toner concentration.

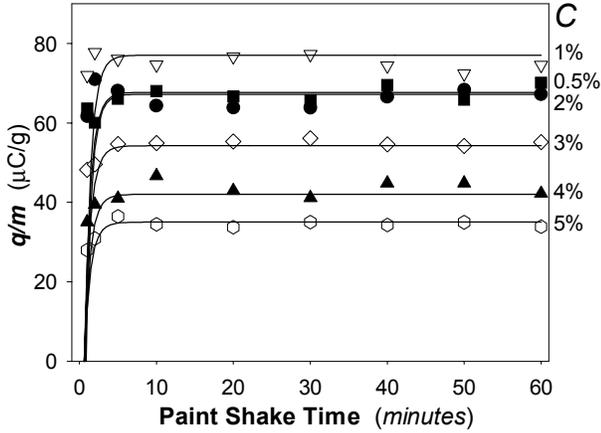


Figure 5. q/m for CCA-free base toner with surface-CCA-coated carrier. (Note that q/m is positive at all toner concentrations).

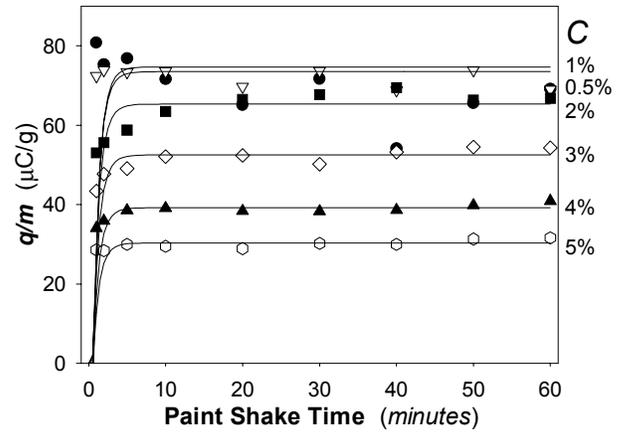


Figure 6. q/m for melt-mixed-CCA toner with surface-CCA-coated carrier. (Note that q/m is positive at all toner concentrations).

Table 4

C wt %	$\phi_{ca,\infty}$ eV	$\phi_{ca,0}$ eV	$\phi_{to,\infty}$ eV	$\phi_{to,0}$ eV	k_{to} min^{-1}	k_{ca} min^{-1}	γ min^{-1}
5	1.50	4.50	4.50	0	2	1	2
4	1.50	4.50	4.50	0	2	1	2
3	1.40	4.50	4.50	0	2	1	2
2	1.60	4.50	4.30	0	2	1	2
1	2.30	4.50	4.10	0	2	1	2
0.5	3.06	4.50	3.60	0	2	1	2

Table 5

C wt %	$\phi_{ca,\infty}$ eV	$\phi_{ca,0}$ eV	$\phi_{to,\infty}$ eV	$\phi_{to,0}$ eV	k_{to} min^{-1}	k_{ca} min^{-1}	γ min^{-1}
5	1.90	4.50	4.50	1.80	2	1	2
4	1.70	4.50	4.50	1.80	2	1	2
3	1.50	4.50	4.50	1.80	2	1	2
2	1.70	4.50	4.30	1.80	2	1	2
1	2.40	4.50	4.10	1.80	2	1	2
0.5	2.90	4.50	3.60	1.80	2	1	2

Test 5: Surface-CCA-treated carrier with melt-mixed-CCA toner

For this carrier/toner combination, the toner contains its own level of CCA and this therefore reduces the tendency for CCA transfer from the carrier surface. As a result, at high toner concentrations the developer based on the CCA-treated carrier and the melt-mixed-CCA toner actually gives a lower positive q/m value than that seen with the CCA-free toner when paired with the CCA-treated carrier.

Figure 6 shows the entire data set, and Table 5 lists the fitted values. For the surface-CCA-treated carrier, the controlling difference between the charging performance of the CCA-free and melt-mixed-CCA toner is the effect of these toners on the value of $\phi_{carrier,\infty}$ (compare Tables 4 and 5). The net result is a CCA loss from the CCA-coated carrier of about 80% at the highest toner concentrations with the melt-mixed-CCA toner (compared with the 90% loss deduced for the CCA-free base toner).

Test 6: Aged carrier with melt-mixed-CCA toner

When aged in a toner throughput imaging test, a carrier is exposed to a continual supply of fresh toner. As a result, in addition to any coating loss, the surface of a carrier may acquire a level of CCA from the toner particles, when aged with a CCA-toner. Figure 7 shows experimental q/m data for an aged version of the test carrier, paired with the test melt-mixed-CCA toner. Basically, this combination gives increased developer stability and a higher positive

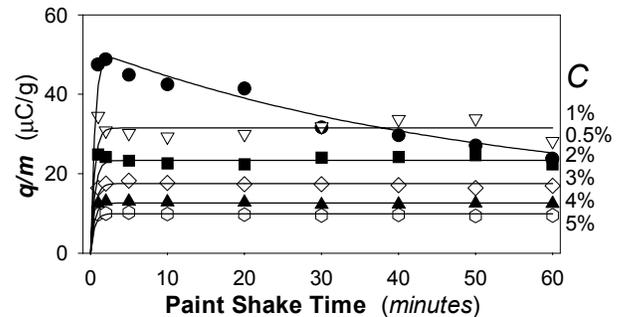


Figure 7. q/m for melt-mixed-CCA toner with aged carrier. (Note that q/m is positive at all toner concentrations).

charging toner over that given with new carrier (e.g., see test 2). Based on a value of 1.8 eV for $\phi_{toner,0}$, the data in Figure 7 yield a value of 0.95 eV for $\phi_{carrier,0}$, with only a minor change in both values throughout the test. Evidently the aged carrier can be viewed as being in equilibrium with the CCA level on the toner surface.

Test 7: Aged carrier with CCA-free base toner

For the combination of an aged carrier and a CCA-free toner, the experimental q/m data shown in Figure 8 appear to show an initial CCA transfer from the aged carrier to the toner, and the deduced $\phi_{toner,0}$ value is close to that of the melt-mixed-CCA toner. (compare this result with that shown in Figure 1, where the q/m values are all negative for the base toner with a new sample of the carrier). At long mixing times, however, the q/m values in test 7 tend towards zero for all toner concentrations, perhaps indicative of a convergence of the values of $\phi_{toner,\infty}$ and $\phi_{carrier,\infty}$ (as a result of an eventual loss of CCA from the toner surface).

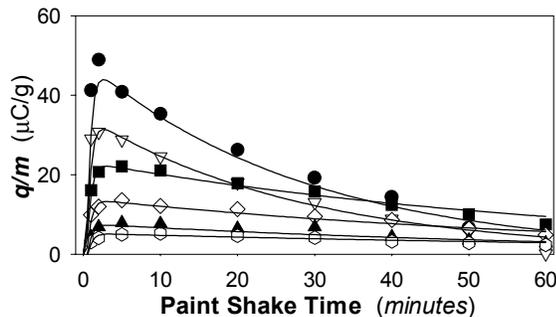


Figure 8. q/m for CCA-free base toner with aged carrier. (Note that the q/m values are all positive at short mixing time, ranging from +40 $\mu\text{C/g}$ at 0.5% to +5 $\mu\text{C/g}$ at 5% toner concentration).

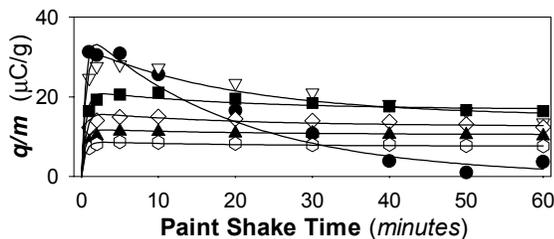


Figure 9. q/m for melt-mixed-CCA toner with a 50:50 mixture of new and aged carrier. (At short mixing times, the q/m values increase regularly in the order of decreasing toner concentration)

Test 8: Melt-mixed-CCA toner with a 50:50 mixture of new and aged test carrier

As shown in Figure 9, an equal mixture of new and aged carrier gives a stable, positive-charging developer with melt-mixed-CCA toner at all toner concentrations greater than 1 wt%. For this case, CCA transfer to the new carrier

can potentially occur from the aged carrier and from the CCA-containing toner, in addition to transfer between the toner and the aged carrier. Overall, there does not appear to be any major change, except at the lowest toner concentrations, and even under such conditions the developer maintains a positive toner polarity. (The net result is similar to that based on a non-interacting 50:50 mixture of new and aged carrier).

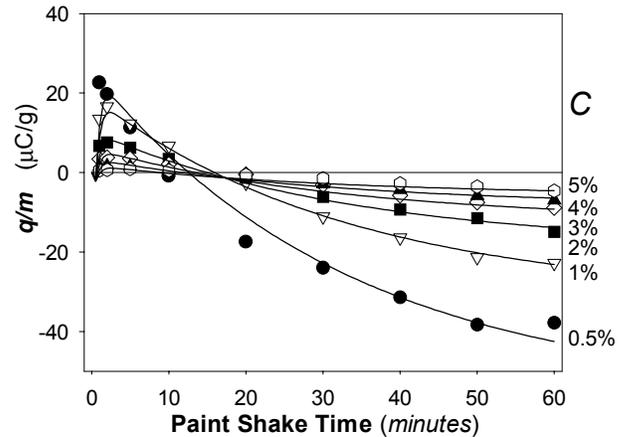


Figure 10. q/m for CCA-free base toner with a 50:50 mixture of new and aged carrier. (Note the common q/m value after 17 minutes of mixing).

Test 9: CCA-free base toner with a 50:50 mixture of new and aged test carrier

In this test, the toner rapidly charges to a positive polarity, but the charge slowly changes to a negative polarity with extended developer mixing. Apparently, continued developer mixing causes a loss of surface CCA from the toner, thus leading to an eventual reversal in polarity. The existence of a common q/m value after about 17 minutes of developer mixing (see Figure 10) indicates that the changes in q/m follow a common mechanism for toner concentrations from 1 to 5 wt%. (The overall effect does not match a prediction based on a non-interacting 50:50 mixture of new and aged carrier — the CCA-free toner clearly receives CCA from the aged carrier).

Summary and Conclusions

From the present experimental data set, it is clear that the charging performance of a toner can be greatly altered through the use of a CCA. However, it is also evident from the data that incorporation of a CCA can create an extremely complex charging behavior.

The tests based on CCA-surface-coated toners and carriers illustrate the major influence that surface-CCA can have on toner charge and on overall charge stability. These tests also reveal that transfer of CCA between toner and carrier surfaces (in both directions) can control the polarity and magnitude of toner charge.

Though complex, the charging behavior of CCA-based developers can be described by a self-consistent simulation model where added CCA alters the charging tendency of the toner and carrier surfaces. In this regard, the influence of CCA on toner charge appears similar to that seen with external additives such as TiO₂ and SiO₂^{5,8,10}. However, a key difference between such latter particulate charge-modifying additives and CCA's is that CCA's may function as surface-active film forming additives, and thereby be especially effective in transfer processes even at low additive concentrations.

Finally, in actual operational conditions, toner particles are continually removed from a developer as a result of xerographic development, with fresh toner being added to counter this loss. As a result, processes such as CCA transfer may be affected by the rate of toner throughput in an imaging developer, and the present data (taken under fixed batch conditions) should probably be only viewed as representative of the extreme condition of zero toner-throughput for any conventional xerographic imaging system.

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Biography

Robert Nash received his Ph.D. in Physical Chemistry from the University of Bristol, England. He joined the Xerox Corporation in 1970. For the past three years he served an expatriate assignment at Fuji Xerox, Takematsu, Japan, as the Senior Manager, Resident for the Xerox Supplies Development, Manufacturing and Supply Chain Operations organization. His research and modeling studies have focused on the design and evaluation of xerographic toners, carriers and developers, with especial emphasis on "aging" mechanisms. Starting with the 4th. International NIP Congress in 1988, he has yearly presented the results of his studies at the IS&T NIP Conference. In 1990, he served as Publication Chairman for the 6th. NIP Congress, and in 1992 he was Chairman of the IS&T Honors & Awards Committee. In 1999, he was named as a Fellow of the IS&T.