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Adhesion of Silica-Coated Toner Particles to Bisphenol-A Polycarbonate Films: Effect of Toner Charge

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Abstract. *Particles of electrophotographic toner (black, silica-coated, number-weighted, and volume-weighted median diameters of 6.5 and* 8.0 μm*, respectively), having two different charge levels (nominally 30 and* 55 μC/g*), were electrostatically developed on the surface of a film of bisphenol-A polycarbonate coated on nickelized polyethylene terephthalate. The thickness of the coatings varied from* 0.8 to 22.0 μm*. The force needed to remove the particles was determined using ultracentrifugation. We found that removal of half of the particles from the film surface required a force of approximately* 200 nN *for all charge levels and film thickness except for the highest charged particles on the thinnest film. These results indicate that, for toner particles of this size, which typically have a charge of approximately* 20–25 μC/g*, adhesion is dominated by van der Waals interactions. However, for highly charged particles, electrostatic interactions can also significantly contribute to the adhesion forces. © 2009 Society for Imaging Science and Technology.*
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INTRODUCTION

The nature of the interactions giving rise to the adhesion of toner particles to the photoreceptor[1–3] in an electrophotographic engine has long been of interest[4–23] and has become a topic of increasing importance as the diameter of the toner particles has decreased from over 20 μm to the present values of less than 8 μm.

Two distinct types of interactions have been proposed to explain the adhesion of toner particles to a photoreceptor. The first assumes that the adhesion arises from electrostatic interactions between the charged toner particle and the photoreceptor. The second proposes that electrodynamic interactions such as those giving rise to the dispersion contribution to van der Waals forces contribute strongly to the adhesion and may often be the dominant force. As discussed by Gady et al.,[24] both types of interactions are present and contribute to toner adhesion. The more appropriate question is which of these types of interactions is dominant and under what conditions. The debate over the dominant mechanism has been quite extensive and a summary has been presented elsewhere.[25]

That both van der Waals and electrostatic forces contribute to toner adhesion has been shown in several recent papers. For example, Rimai et al.[25] showed that the force needed to remove a 7 μm diameter spherical toner, without a silica surface treatment, from a standard commercial organic photoreceptor that had not been overcoated with any release agent such as zinc stearate was in excess of 750 nN, which was the maximum force they could exert. They estimated from Johnson–Kendall–Roberts (JKR) theory[26] that the van der Waals force alone would be in the range of 1100 to 1200 nN. They also found that, by applying zinc stearate to the surface of the photoreceptor, the van der Waals forces were reduced to approximately 100 nN and the remaining detachment force varied as the square of the charge on the toner and that, at sufficiently high charge (in excess of that possessed by toner particles in a normal developer), electrostatic forces dominated the adhesion.

Van der Waals interactions have a range in the order of only a few nanometers, whereas electrostatic interactions have a much longer range. Dejesus et al. used these facts, as reported in a recent paper,[27] to attempt to distinguish the dominant interaction for a commercially available silica-coated ground toner having a median number-weighted diameter of 7.1 μm and a charge-to-mass (q/m) ratio of −24.3 μC/g. In that paper, they reported detachment forces, measured using ultracentrifugation, for removal of the toner particles from nickelized polyethylene terephthalate (PET) that had been overcoated with bisphenol-A polycarbonate films ranging in thickness from 0.8 to 22.0 μm. They found that the detachment force was approximately 300 nN and was independent of the thickness of the polycarbonate layer. Moreover, using a corona to spray positive charge onto the surface of the polycarbonate did not appear to affect the detachment force. In their analysis, they concluded that van der Waals forces, rather than electrostatic interactions, dominated the adhesive forces. Dejesus et al. considered possible contributions from van der Waals interactions, electrostatic forces associated with both uniform charge and localized charged patches,[15] and the so-called "proximity force" model proposed recently by Schein.[28]

Particulate nanoclusters are often appended to the surfaces of toner particles to decrease adhesion.[22] Accordingly, if van der Waals interactions are sufficiently small and/or if electrostatic interactions sufficiently large, then one should be able to detect the contributions to toner adhesion that are due to the presence of the charge on surface-treated toner particles. In the present study, we have expanded on the results reported by Dejesus et al.[27] Specifically, we have mea-

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sured the detachment force required to remove toner particles having relatively high charge from a series of substrates comprising nickelized PET that had been overcoated with bisphenol-A polycarbonate films ranging in thickness from 0.8 to 22.0 μm.

EXPERIMENTAL

In this study, ultracentrifugation was used to determine the force needed to remove highly charged toner particles from a series of substrates comprising nickelized polyethylene terephthalate (PET) overcoated with varying thicknesses of bisphenol-A polycarbonate.

Two developers were prepared by tumbling the toner and carrier on a roll mill for 30 min. These developers comprised the same toner particles but different carrier particles. The toner was a black commercial polyester toner having a median number-weighted diameter of 6.5 μm, as determined using a Coulter Multisizer, and a mass density of 1.2 g/cm^3. Assuming that the particles were spherical, the mass of each toner particle was calculated to be 1.73×10^{-10} g. The toner particles also comprised 1.2% by weight of Degussa Aerosil® R 972 silica applied on the surface. It should be noted that conditions related to surface treating toner particles with silica in a lab differ from those in production environments. These differences include the size and energy input in the blending equipment, aging effects, resulting surface concentrations of silica particles, etc. A detailed discussion of the effect of such variations is beyond the scope of the paper. Suffice it to say, it would be anticipated, and in fact observed in this paper, that the detachment force of these toner particles under nominal baseline conditions (e.g., from substrates comprising thick polycarbonate coatings) will differ from those of the earlier study by Dejesus et al.[27] Otherwise, the toner particles were very similar to those used in the study by Dejesus et al. Although the toner particles in both developers were more highly charged than the toner particles in the commercially available developer used in the study by Dejesus et al., one of the developers was designed to impart a significantly higher charge on the toner particles than the other. The terms "low" and "high" are used to reflect the charge on these two materials.

The carriers were derived from the commercial $SrFe_{12}O_{19}$ hard ferrite core (Powdertech) used in the Kodak NexPress and Digimaster printers. The carriers are prepared by mixing fine polymer beads of Kynar 301 (Arkema) and Soken 1201 poly(methyl methacrylate) with the ferrite core, following by heat treatment to fuse and flow the polymers. The high charging developer comprised a carrier coated with 0.3 pph of poly(methyl methacrylate) and was blended with the toner to give a toner concentration (TC) of 6 wt. %. The low charging developer comprised a carrier coated with 0.45 pph Kynar 301 and 1.80 pph poly(methyl methacrylate), and blended with the toner to yield a 10% TC. The roll mill not only served to mix and charge the toner particles, but also conditioned the toner to simulate developer in an actual development station in a manner consistent with the manufacturing process for commercially available developer.

The charge-to-mass ratios of the low and high charged toner were approximately −35 and −55 μC/g, respectively, as determined using the method described by Maher.[29] These values correspond to particle charges of approximately -6.06×10^{-15} C and -9.52×10^{-15} C, respectively. The toner charge-to-mass ratio was determined before and after deposition onto the substrate and was found not to change during the course of an experiment. It was found, however, that the charge-to-mass ratio changed from day to day, necessitating its determination for each run. The measured relative humidity and temporal variations did allow the effects of charge on adhesion to be determined on a semicontinuous basis. For comparison, the toner particles used by Dejesus et al. had a charge-to-mass ratio of approximately −24.3 μC/g, corresponding to a particle charge of approximately -5.35×10^{-15} C. It should be noted that the toner particles used by Dejesus et al. were approximately half a micrometer larger than the ones used in this study. Fresh developer was used for each experiment.

The substrates comprised nickelized PET that had been coated, from solution, with four different thicknesses of bisphenol-A polycarbonate. The polycarbonate was chosen because of its high electrical resistivity (greater than 10^{16} Ω cm) and low dielectric constant (approximately 3).[30] These values ensure that the dielectric relaxation time of the substrates is very long compared to the duration of the experiment. The coating thicknesses, determined from cross sections using optical microscopy, were approximately 0.8, 4.5, 9.0, and 22.0 μm.

The film sample (approximately 3 in. × 6 in.) was held by vacuum to a grounded aluminum platen and had an edge coated with electrically conducting paint to ground the nickel layer. The platen was positioned over an electrostatic voltmeter and the surface potential of the polycarbonate coating was measured. The coating was then repeatedly washed with isopropanol and blown dry with canned air until the surface potential was zero.

A very light coating of toner particles was deposited on the substrate using a sumpless magnetic brush[2] biased at approximately −10 V. The development system was set up so the flow of the developer was matched to the speed of the sample. This was accomplished by adjusting the magnetic brush shell speed to counter the core speed until there was zero net flow of developer, and then adjusting the shell speed so that the speed of the developer matched that of the substrate. Development under these conditions resulted in a sufficient number of negatively charged toner particles to permit good statistical counting, but still left the particles generally isolated from each other. To verify that the toner deposition and resulting adhesion were not influenced by a triboelectric interaction between the toner particles and the polycarbonate, the discharged substrate was developed in a normal manner with the exception that the brush was also grounded. After passing over the development station, any deposited toner was removed from the substrate using an air knife and the potential on the substrate again measured. Voltages were found to be zero, within the limits of the measurement, thereby ensuring that no significant tribocharging had occurred.

A sample was developed with toner as described and then cut into sections approximately 1/8 in. × 1 in. The number of particles on a sample of the substrate was determined by counting the particles in five areas under a microscope using Image-Pro particle-counting software. The detachment force was determined using a Beckman L8-70M ultracentrifuge capable of speeds up to 70 000 rpm (at 70 000 rpm the acceleration of the centrifuge with the 6.45 cm radius rotor is 354 000 g). The sample was then placed in the rotor and spun at the desired speed for 1 min. The sample was then removed and the number of residual particles was determined in five areas.

In order to eliminate the possibility of increases in adhesion caused by the occurrence of plastic deformations[31] or from the rotation of the particles on the substrate following deposition,[32] all measurements were made on the same day as the deposition. Moreover, the centrifuge speeds were randomized to ensure that any systematic deviation would be observed. The applied force needed to remove 50% of the particles initially on the substrate was considered to be the detachment force.

RESULTS

Figure 1 shows the percentage of the toner particles removed as a function of the applied force for each of the four substrates for both the low- and high-charged toner particles.

Using Fig. 1, one can compare the percent of the particles removed as a function of the applied force. It is seen that half of the particles were detached in all cases, with the exception of the highly charged particles on the thinner samples, at an applied force of approximately 200 nN. The spread in the data is approximately 10% and is random with respect to both particle charge and substrate thickness. This implies that the variations are random and due to the noise of the measurements rather than arising from the deviations in either particle charge or substrate thickness. Sources of the noise can include differences in particle size, shape, silica coating, contact area, and charge. In contrast to the general results, the 0.8 μm thick polycarbonate substrate with high charge toner required greater detachment forces for toner removal. However, results for 4.5, 10, and 22 μm thick substrates show no significant effect due to toner charge.

The detachment force is normally defined as the force at which half of the particles are removed from the substrate. In this study, the detachment force was determined by first plotting the individual curve for the percent removed as a function of the applied force. The scale, centered around the data where 50% of the particles were removed, was expanded to improve resolution and a smooth curve drawn through the data. The force at the point where 50% of the particles were removed was determined directly from that curve.

The force at which 50% of the toner particles were detached was found to be approximately 200 nN for all but the highly charged particles on the thinnest substrates. This force is substantially lower than the value of 300–350 nN reported by Dejesus et al. Part of the apparent discrepancy is due to the fact that the particles used by Dejesus et al. were larger and, therefore, were expected to have a larger detachment force. However, most of the apparent discrepancy most likely results from the differences in the silica concentration and embedment into the toner particles due to the fact that the present set of toners and developers was prepared in a lab, whereas the earlier set comprised commercially available material. This was discussed earlier in this paper.

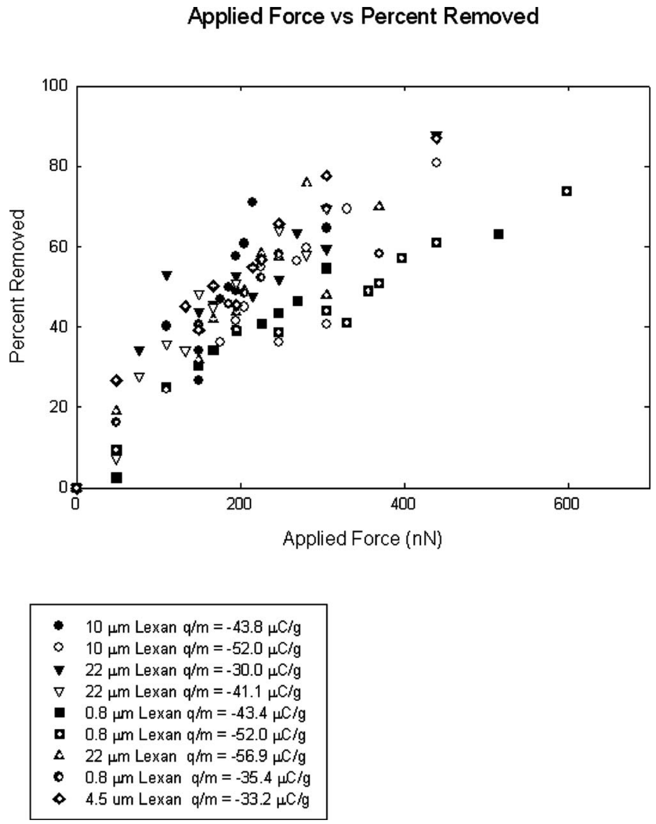

Figure 1. The percent of particles removed as a function of the applied force for each of the four bisphenol-A polycarbonate thicknesses for both the low- and high-charged toner particles.

As the adhesion of silica-coated toner particles is sensitive to the conditions under which the toner particles and developers are made and used, relative comparisons between this and the former study can be made without expecting detachment forces for the two materials to be exactly the same. Suffice it to say, one must be careful when measuring detachment forces of surface-treated toners to make sure that a single batch of toner is used throughout the experiment and that process conditions are kept constant. Such variations can, at least in part, account for the discrepancies in detachment force values reported in the literature.

Figure 2 shows the detachment force as a function of polycarbonate thickness for toner particles having charge varying from −30 to −56.9 μC/g. As is apparent, the detachment force appears, in general, to be independent of the thickness of the polycarbonate coating. The only clear dis-

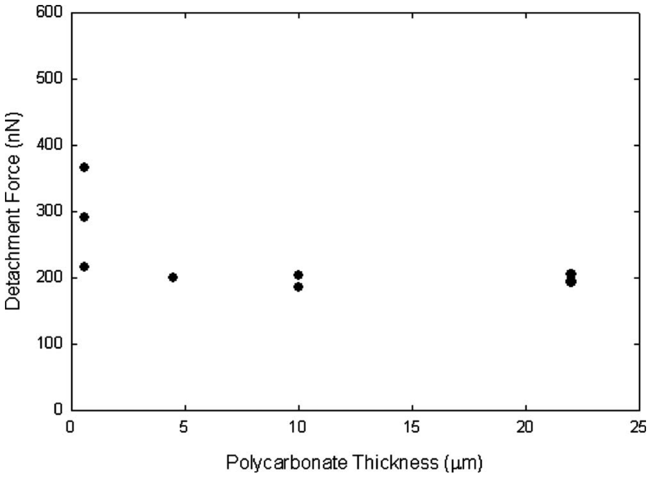

Figure 2. Detachment force as a function of polycarbonate thickness.

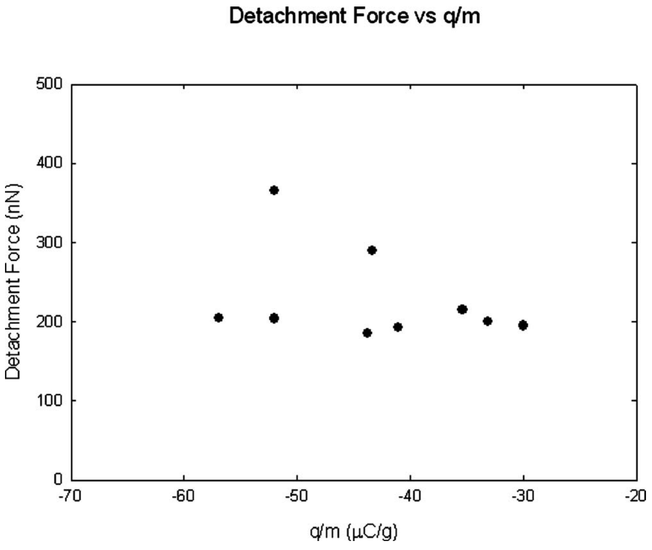

Figure 3. Detachment force as a function of *q*/*m*.

crepancies to this are observed for two values of approximately 290 and 360 nN, corresponding to q/m values of -43.4 and -52 μC/g, respectively, on the thinnest substrates used; i.e., 0.8 μm thick polycarbonate. It should be noted that there are actually three data points for the 22 μm thick polycarbonate substrate, with the third point being covered by the other two.

Figure 3 shows the detachment force as a function of the toner charge-to-mass ratio. It is apparent that the detachment force is independent of q/m with the exception of two outliers. These outliers, as was the case in Fig. 2, correspond to the two highly charged toners on the 0.8 μm thick polycarbonate coating.

Figure 4 shows a three-dimensional graph depicting the effect of charge-to-mass and polycarbonate thickness on the detachment force. As is apparent, the detachment force is independent of both q/m and polycarbonate thickness except for the two highly charged toner samples on the thinnest (0.8 μm) polycarbonate.

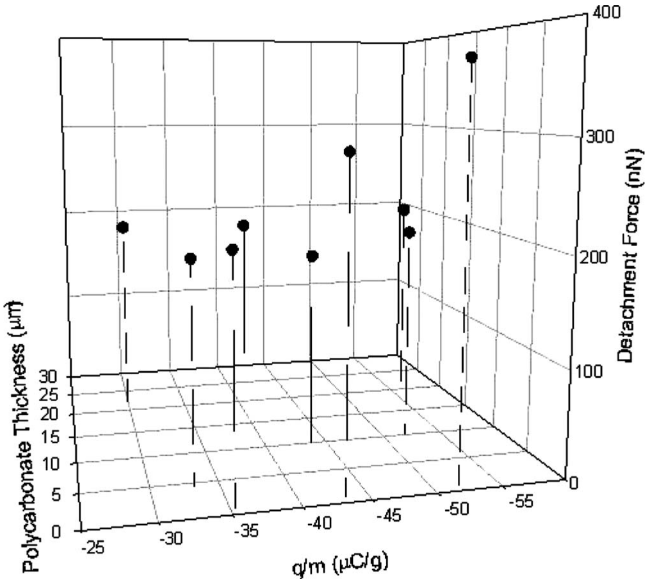

Figure 4. The effect of *q*/*m* and polycarbonate thickness on the detachment force.

DISCUSSION

As mentioned earlier in this paper, theories of toner adhesion focus on two types of interactions: van der Waals forces and electrostatic forces. Of these, the former have a range of a few nanometers, whereas the latter are longer range. Accordingly, all of the polycarbonate coatings used in this study were infinitely thick as far as van der Waals interactions were concerned. However, the films could be considered as ranging from thin to thick for electrostatic interactions. A detailed analysis of the effect of the thickness of the polycarbonate film on the electrostatic contribution to adhesion has been presented in an earlier paper.[27] Suffice it to say, if electrostatics play a major role in the adhesion of the toner particles, a decrease with coating thickness and increase with toner charge would be expected. Depending on whether one assumes a uniformly distributed charge over the surface of the particle or, alternatively, the extreme case of the charge patch model where all the charge is located at the particle-substrate contact, the electrostatic force would be expected to decrease by as much as three orders of magnitude.

As discussed by Dejesus et al.,[27] van der Waals forces can readily account for the adhesion of toner particles to a photoreceptor. Specifically, the detachment forces predicted by JKR theory[26] predict a detachment force for spherical toner particles in the present size range of approximately 1100 to 1200 nN. Moreover, the decrease in the detachment force resulting from the silica coating on the surface of the toner particles is readily explained by the model of Fuller and Tabor,[33] which modified the JKR model to treat the role of asperities. This topic has been discussed elsewhere[22] and further discourse is beyond the scope of this paper.

It is clear from Figs. 2 and 3 that neither the thickness of the dielectric layer separating the charged particle from the nickel layer nor the toner charge affects the detachment force except for the most highly charged particles on the 0.8 μm polycarbonate film. It would appear from these results that approximately 200 nN of the total force of adhesion could then be attributed to van der Waals interactions, which would be expected to depend neither on the particle charge nor on the thickness of the polycarbonate coating.

Feng and Hays[34] have calculated the effect of an intervening dielectric material on the electrostatic force between a charged particle and a conductive ground plane. They report that large changes will occur when the thickness of the dielectric is less than the particle diameter. We do not observe such an effect, particularly for toner particles with charges in the mid-30 μC/g range, with the possible exception of the thinnest of the dielectric layers and the highly charged particles.

Let us now explore possible mechanisms relating to the increase in adhesion observed for the highly charged particles on the 0.8 μm polycarbonate coated nickelized PET.

The effect on adhesion of a charge distributed uniformly over the surface of a particle has been often demonstrated to be inconsequential. Accordingly, no further discourse on that topic will be presented in this paper. Rather, it would be more fruitful to concentrate on effects such as those attributed to localized charged patches.[35] According to the model proposed by Hays, the force of attraction F_A between a nonuniformly charged particle and a conducting substrate is given by

$$F_A = \frac{\sigma^2 A_C}{2\varepsilon_0}, \tag{1}$$

where σ, A_C, and ε_0 represent the surface charge density, the contact area between the particle and the substrate, and the permittivity of free space, respectively. However, Eq. (1) cannot be used to calculate the charged-patch contributions in the present instance because: (A) it assumes that the contact area is sufficiently large that parallel plate geometry can be assumed and (B) it does not allow for an intervening dielectric material. The latter topic has recently been explored by Crowley.[36]

It is perhaps more advantageous to calculate the charge needed to give the incremental force over and above that which we attribute to van der Waals interactions, assuming that the charge lies at the point of contact between the particle and substrate and not allowing for the materials to deform. Accordingly, the electrostatic force is given by

$$F_A = \frac{1}{4\pi\varepsilon}\left(\frac{q_{\text{point}}}{d}\right)^2, \tag{2}$$

where ε is the relative dielectric constant of the polycarbonate (approximately 3) and d is twice the thickness of the polycarbonate film.

Rewriting Eq. (2) so as to solve for q_{point}, one finds

$$q_{\text{point}} = [4\pi\varepsilon d^2 F_A]^{1/2}. \tag{3}$$

Assuming that $d = 1.6$ μm (twice the thickness of the thinnest of the polycarbonate coatings) and that the incremental force F_A is approximately 150 nN, one finds that $q_{\text{point}} = 11.3 \times 10^{-15}$ C. This is comparable in magnitude to the total charge on the particle, which was calculated to be -9.5×10^{-15} C. Polarization effects, as discussed by Crowley,[36] exist and can contribute to particle adhesion. However, such effects are expected to be large only when the dielectric constants of the materials are large. It is highly implausible that the enhancement to the adhesion of highly charged toner particles to a thin organic photoreceptor can arise entirely from a localized charge distribution. Specifically, in both this and a previous study,[27] electrostatic contributions to the detachment force were only observed for those particles possessing a very high charge on the 0.8 μm thick polycarbonate. In comparison, toner particles normally used in electrophotographic engines have lower charges, comparable to those reported by Dejesus et al.[27] Moreover, organic photoreceptors are typically 20–25 μm thick—comparable to the thickest polycarbonate coating used in this study. Thus, we can conclude that the predictions of adhesion models that rely on simple electrostatic interactions, such as the charged patch model, appear to be inconsistent with the observed effects for the materials studied.

If the measured detachment forces are not consistent with simple electrostatic models, what can account for the observed charge dependence of the detachment force for the higher charged toner particles from the 0.8 μm thick polycarbonate film? Two possible models suggest themselves. The first assumes the presence of both positive and negative charges on the surface of the toner particles, giving rise to a net charge as measured. As the force of attraction of the particles to the substrate depends solely on the magnitude of the charges and not on the sign of the individual charges, an enhancement above and beyond the expected effects of net charge can occur. This hypothesis, however, begs the question as to why similar effects are not observed on the lower charged particles used in either this study or the previous work by Dejesus et al. Moreover, evidence of such a charge distribution on toner particles is not generally observed. It might occur on developers that contain wrong-sign and toner particles with low charge-to-mass ratios, but these usually result in development artifacts such as dusting, indicating that low charge developers do not have enhanced adhesion due to the presence of charges of both polarities.

An alternative hypothesis to explain the increase in adhesion observed for the highly charged particles on the thin polycarbonate coating presupposes that electrostatically induced torques on the toner particles, which are greater because the charges on the particles are closer to the ground plane, cause them to rotate into a more adhesive position. Indeed, such an effect has been reported in the literature by Dejesus et al.,[37,38] who observed such an effect with similar, commercially available toner particles on ceramer-coated nickelized PET. In those cases, however, the particles had a lower charge-to-mass ratio and the increase in adhesion took several hours to occur. Any such temporal effect would have been observed in the present study, as the randomization of the speeds at which the centrifuge was run would have resulted in a systematic drift toward higher adhesion during the course of the experiment. Such a drift was not observed. Therefore, the rotation would have had to occur over a time scale of, at most, minutes. This might be possible, given the higher charge of the particles in which the increase in adhesion was observed. Further research is needed to determine if either of these hypotheses is valid.

CONCLUSIONS

We have found that the force required for removal of the silica-coated toner particles (median number-weighted diameter of approximately 6.5 μm) from a film of bisphenol-A polycarbonate on nickelized PET is approximately 200 nN and is independent of the thickness of the coating, except in instances of very highly charged toner particles deposited onto thin (0.8 μm) films. For the case of very highly charged particles on very thin polycarbonate films, toner adhesion was observed to increase with charge. The results suggest that, for the toner particles used in this study, adhesion is dominated by van der Waals forces rather than by electrostatic interactions. Further work is planned to differentiate between electrostatic and van der Waals forces for comparable toner particles having lower detachment forces.

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