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Effect of re-use of surface sampling traps on surface structure and collection efficency for trace explosive residues

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ABSTRACT

In security settings, explosive residues or particles are collected by swiping the object of interest (e.g., luggage or package) with a collection medium, or trap. Particles on the trap are thermally desorbed for detection by ion mobility spectrometry (IMS) or other analyses. A high trap sampling efficiency increases the chance of detection, and is affected by a number of factors. In particular, this work studies the effect of trap re-use on collection efficiency of organic explosives, namely 2,4,6-trinitrotoluene (TNT) and 1,3,5-trinitro-1,3,5-triazinane (RDX), and correlates this data to quantifiable morphology changes. Collection efficiency was measured by liquid extraction of the traps with detection and quantitation by gas chromatography / mass spectrometry (GC/MS). Using silhouette microscopy for visualization of the trap texture, morphology changes were quantified by several measurements of trap roughness and hairiness, drawing from techniques and metrics used in the textiles industry.

Nomex traps were visibly roughened by repeated re-use, and this was correlated with significant improvements in trap collection efficiency (11–57%) depending upon the specific analyte and substrate combination interrogated. Teflon-coated fiberglass (TCFG) traps showed little change with repeated swiping and minimal to no improvement in particle collection efficiency. These results have direct implications for optimizing particle collection traps for use in security settings.

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1. Introduction

A variety of explosive detection technologies are utilized to prevent explosives from entering secured areas such as transportation or shipping facilities, ports and borders, government buildings, landmarks, embassies, and nuclear and chemical plants. Such technologies encompass a large range of instrumentation to include both bulk and trace detection. Bulk detection methods are often imaging or scanning techniques that allow the user to see a form that is suggestive of an explosive device, while trace detection methods use chemical analysis to identify specific chemical components indicative of explosive materials. Ion mobility spectrometry (IMS) is the most common analyzer used in explosive trace detectors (ETDs) both for homeland and military security purposes.

Trace explosive residues are generally delivered to a commercial IMS system in the form of explosive particles by means of swiping explosive residue from an area of interest with some sort of sampling swab or trap. Vehicles, luggage, packages, or persons are often sampled for explosive residue. For the sampling of trace residue to be successful, (1) a detectable quantity of explosive material must be left behind after contact between the explosive material and another surface of interest (i.e. residue transferred to the hand then to a touched surface); (2) the sampling procedure must effectively remove the residue and transfer it to an analyzer; and (3) the analyzer system must effectively vaporize, ionize, detect, and correctly identify the explosive material. To address the first concern, research has shown that a detectable level of explosive residue is indeed transferred following handling of the material [1,2]. Verkouteren et al. showed that RDX particles from C-4 residue could be detected on a glass surface even after 50 consecutive fingerprints [1]. Transfer directly onto substrates has also been presented by Tam et al., who directly transferred quantifiable C-4, Detasheet, Semtex-H, TNT, and HMTD residues onto different surfaces [3].

To address the third point, it is known that desorption and subsequent vaporization is highly dependent on IMS inlet temperature, as certain explosive analytes (such as PETN)

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dissociate at relatively low temperatures, while other low volatility analytes (such as RDX) require a higher temperature to desorb and be vaporized. For this reason, trap materials are chosen for their ability to withstand IMS desorber temperatures of 100 °C – 300 °C while also delivering minimal background signal for IMS. Detection efficiency is also dependent on particle placement, homogenous trap heating, and efficient vapor transport [4].

The focus of this work is on the second point. Without suitable sampling methods explosive residues cannot be efficiently transferred from the object of interest to the detection system, and if no explosive residue is collected during sampling, the performance of the detection system is irrelevant.

Residue swiping exploits downward pressure and friction forces to remove particles of interest from the sampled substrate. The capture of a particle can be considered as a simple balance of forces. For a particle to be removed from a substrate, adhesion forces between the particle and substrate must be overcome by forces between the particle and the trap. A majority of the adhesion force is due to van der Waals interactions, but electrostatic, capillary, and friction forces can also play a role [5–7]. The magnitude of the adhesion force is affected by both the substrate and analyte chemical composition and by morphology [7].

A number of factors affecting swipe efficiency have been investigated, including the impacts of trap / substrate material (chemical and morphological), swiping force and speed, analyte chemical structure, and particle size [5,7–9,3,10,11]. An increase in particle collection efficiency has been noted when the collection trap has a surface texture incorporating long, unsupported fibers [5], or when the trap has been roughened [11,10]. These results could be explained by an increase in the total surface area available for particle binding on roughened trap surfaces, or perhaps by a greater likelihood of particle entanglement within a matrix of loosened fibers. Meanwhile, increased surface roughness of the substrate generally reduces the detachment force for any individual particle [12–16], an effect attributed to a smaller particle/substrate contact area. These effects can be sizable: for example, Yu et al. [16] found that the average force required to remove a 10-μm-diameter TNT particle from a smooth glass surface was more than five times higher than the force required for a rough mercerized cotton surface.

This work studied the effect of trap material wear (due to re-use) on collection efficiency. In high-throughput security check points, such as airport security, swiping materials are often re-used following a negative result to minimize the cost of consumables, though this practice is not necessarily recommended by the manufacturer. Several research groups have assessed the viability of the re-use of swiping materials. Both Staymates et al. [11] and Fisher et al. [10] investigated the effect of trap re-use on collection efficiency. Both groups used scanning electron microscopy (SEM) to evaluate changes in trap morphology with continued re-use. While both studies agreed that collection efficiency was improved with re-use, Fisher et al. showed a decline in collection efficiency after 5–10 successive uses, while Staymates et al. indicated a continued increase in particle collection efficiency after as many as 1000 re-uses.

The discrepancy between the two studies could be due to the explosive particles used in the interrogations. Staymates used polystyrene latex (PSL) spheres as surrogates for explosives particles, while Fisher used HMX and RDX explosive residues. In addition, Staymates conducted their sampling in a controlled laboratory setting while Fisher elected for a more realistic (but less controlled) test in an airport screening setting.

The study described herein expands upon the previous research by including additional explosive materials and substrates in the investigation of the effect of trap re-use on collection efficiency, while controlling sampling parameters in a laboratory setting. Analytes studied in this work included TNT (not often considered due to its higher volatility) and RDX from solution. In addition, RDX particles imparted from C-4 residue were also chosen, as RDX is rarely detected alone and it was hypothesized that the plasticizers in the C-4 residue would dramatically affect the adhesion of the analyte to both the substrate and the trap. Collection efficiency and wear due to re-use was evaluated with two field-relevant materials encountered in airport and cargo security check points. For this purpose Cordura, a nylon-blend fabric commonly used in backpacks and luggage, and kraft paper used in cardboard were considered as substrate materials. Collection materials investigated were commercial Nomex and Teflon-coated fiberglass (TCFG) traps. These are two of the most common trap materials used with IMS instrumentation. All experimental parameters including explosive residue deposition, trap roughening, sampling, and analytical quantitation were closely controlled and validated to ensure any observed variations were genuine and not due to variability inherent in field-sampling scenarios.

Furthermore, for the first time, this study correlates collection efficiency results from swiping experiments to quantifiable morphology changes in the traps measured using a silhouette microscopy and image analysis technique. Though interactions of particles with micro-scale features of the collection trap and substrate play essential roles in particle capture, very little has been published regarding best practices for visualizing, quantifying, and comparing the surface textures of swipe-sampling traps. Drawing from work in textiles, we show here that silhouette image analysis [17,18] is particularly well-suited to this application. In this method, the textile's surface is observed from the side using a horizontally-mounted microscope, and surface roughness is evaluated through image processing. This technique allows loose fibers, pills, and fuzz to be evaluated separately from the dense fabric layer below using image segmentation and multilayer analysis. These are then quantified using techniques from the textile industry as trap 'hairiness' [19,20] and roughness.

2. Materials and methods

For each trap material, varying levels of re-use were simulated by swiping the trap across an explosives-free substrate material (Cordura or paper) 0, 20, or 100 times. Following this preparation, traps were swiped across a substrate dosed with a known quantity of explosive residue. Traps were then analyzed to determine the trap collection efficiency using solvent extraction. All samples were prepared in replicates of five for the swiping experiments; a separate set of five replicates per trap/re-use condition were prepared and used for texture evaluation.

2.1. Materials

Two Department of Homeland Security (DHS)-approved swipe-sampling materials were used in this work, including a nonwoven meta-aramid (Nomex®) sample trap (DSA Detection, Part No. DSW8066) and a woven polytetrafluoroethylene- (Teflon®-) coated fiberglass (TCFG) sample trap (DSA Detection, Part No. ST1322). For clarity, we refer to the products used in these experiments individually as Nomex or TCFG, respectively, and collectively as traps.

Sample trap materials were used to recover trace explosives from two substrates, Cordura (0.02 inch thickness; McMaster-Carr Supply Co., Part No. 8809K31), a material often used in luggage and backpacks, and kraft paper (Brown Kraft Wrapping Paper), representing the outer layer of cardboard packages. The textured side of the Cordura, the side usually facing outward in luggage, was used exclusively. Both types of substrate were backed with corrugated cardboard (cut from ordinary shipping boxes) for resiliency and flatness.

The explosives tested were 2,4,6-trinitrotoluene (TNT), 1,3,5-trinitro-1,3,5-triazinane (RDX), and Composition C-4 (C-4). TNT and RDX were obtained as 10 mg/mL solutions in acetonitrile (AccuStandard, Inc.). C-4 was provided by the Federal Bureau of Investigations, Explosives Unit (FBI-EU) and contained approximately 88–90% RDX.

2.2. Sample trap preparation

A mechanical wiper capable of simultaneously swiping ten traps across a surface was used to reproducibly emulate the re-use of trap materials in a manner representative of typical swiping by human operators [9] and imitating protocols previously developed by Staymates et al. [11]. Swiping was automated using a computer numerical control (CNC) milling machine (repurposed for its motion base) and a custom swiping fixture that accommodated ten traps. The fixture comprised a square plate with holes drilled to accommodate an array of 1.25-inch-diameter (31.8-mm-diameter) steel clevis pin "tups" (McMaster-Carr Supply Co., Part No. 98,306A851). A circular piece of adhesive-backed, 0.125-inch-thick (3.2-mm-thick), medium-hardness silicone sheet (McMaster-Carr Supply Co., Part No. 1464N14) was attached to the bottom of each tup to mimic the compliance of a handheld swiping wand. Each trap was secured against the silicone base of the tup using a nylon cross bolt and a cable tie. Photos of the mechanical swiping fixture are provided in Supplementary information.

The downward swiping force was maintained at 7 N and the overall travel was 100 mm for each swipe. Downward swiping force was provided by the mass of the tup, which was allowed to move freely along the vertical axis. For each swipe, the fixture holding the traps was lowered onto the test substrate, and the substrate was translated laterally under the fixed trap array at a rate of 30 mm/s. The trap fixture was then raised and the machine stage was returned to its original position to perform the next swipe. Note that the clevis pins were arranged in the fixture such that there was no overlap between swipe paths of individual traps. Traps were swiped across either of the two substrates to simulate re-use (no particles). The Kraft paper and Cordura substrates were replaced after every 20 swipes to minimize the effect of substrate wear.

2.3. Imaging and measurement of trap surface texture

Trap texture measurements were made for five replicates of each combination of trap / re-use condition. A horizontally-mounted zoom lens with a 165-mm working distance (Navitar Inc., 12× Zoom Lens System), equipped with a machine vision adapter (Navitar Inc., Model 1-6233) and Gigabit Ethernet camera (Prosilica, Model GC750), was used to capture "silhouette" images of the trap's surface texture (Fig. 1). A blue LED backlight aligned with the optical axis of the zoom lens was used for illumination; this color is preferred for monochromatic silhouette imaging as short-wavelength visible light produces the highest-resolution images. Magnification settings available in the zoom lens ranged from 0.58× to 12.0×, and the 1.0× setting was selected for image processing as this setting provided visualization of the largest area on the sample while still providing adequate resolution of trap surface texture. Captured images show the trap and unsupported surface fibers as a dark silhouette on a light background. Traps were secured to a curved mount using double-sided adhesive tape, isolating a region across the center of the trap for imaging and keeping the trap's edges out of view to avoid effects of edge fray.

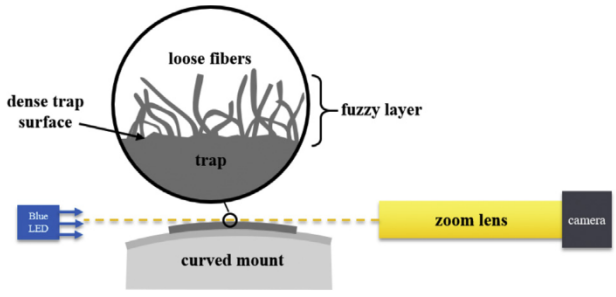

Fig. 1. Schematic of silhouette imaging technique for particle collection traps. (For interpretation of the references to color in the text, the reader is referred to the web version of this article.)

A semi-automated image-processing algorithm was developed using the ImageJ image analysis software package. The algorithm isolates loose surface fibers on the trap surface as a binary image through bi-thresholding and mask creation, as shown in Fig. 2. The upper boundary of this binary image defines the surface profile of the "roughened layer" of loose surface fibers, while the lower boundary of the binary image defines the surface profile of the trap's "dense layer" below. Treating these two profiles separately in roughness measurements allows for contributions of the surface fibers to be distinguished from the overall trap texture. This was found to be important for useful comparisons between textile traps, since surface fibers were present on some samples but not all, could be long or short, and exhibited varying levels of entanglement with one another. Additional details of the image-processing algorithm are included in Supplemental Information.

Six texture parameters, summarized and defined in Table 1, were evaluated for each image using the image-processing algorithm. The first four parameters (*total roughness* R_A and *roughness average* R_T) are conventional measures of roughness and were measured separately for the dense and roughened layers of the trap. The silhouette microscopy technique is unique in enabling roughness measurements for both of these layers. Note that in comparing to other work, values for the dense layer could be reasonably compared to conventional contact profilometer measurements (where unsupported fibers are compressed by the profiler during measurement), whereas roughness measurements for the roughened surface layer could be reasonably compared to measurements made using a conventional noncontact optical profilometer (where unsupported fibers would be included in the surface profile).

The *roughened layer thickness* T_R is defined as the maximum perpendicular extension of a surface fiber from the dense trap surface, and is useful in evaluating the total extent of this layer. Finally, the *hairiness index* H_L is defined as a dimensionless ratio of the cumulative length of loose surface fibers to the sampling length. The concept of a "hairiness index" is drawn from the textiles industry, where it is used to describe tactile properties or quality of yarns [19,20]. In that industry, the use of the hairiness index is so widespread that it has given rise to dedicated measurement equipment (e.g., the Shirley yarn hairiness tester). However, surprisingly this metric is generally used only for one-dimensional yarns, and to our knowledge the technique has not been widely extended to other applications (e.g., two-dimensional textiles) or other industries. We find it to be useful for evaluating the extent of loose fibers in the surface texture of traps, and the definition is readily adaptable for the two-dimensional trap surfaces used in this work.

For each trap, the six texture parameters were measured in five separate locations; results were averaged to calculate the texture parameters for that trap sample. Following measurement of all individual traps, statistics were computed for all replicates of each treatment group.

2.4. Quantitative analysis of surface wipe efficiency

The dry transfer method [21] was utilized for the deposition of RDX and TNT residues onto test substrates. This method was

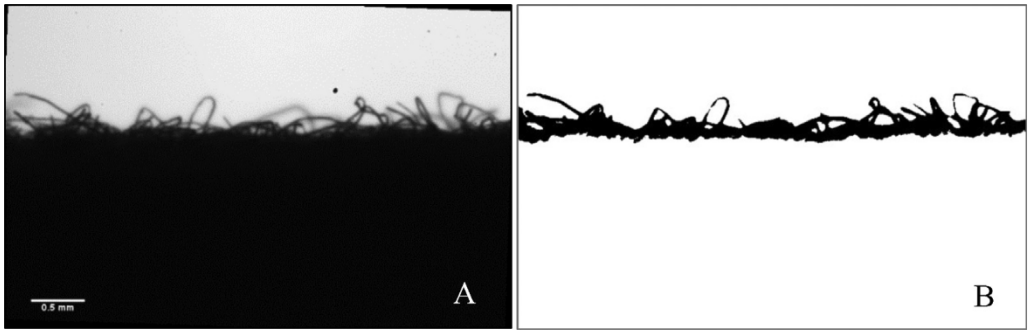

Fig. 2. Example of binary mask creation for a Nomex trap image: (A) original image; (B) binary mask isolating the roughened surface layer.

Table 1
Trap texture parameters evaluated.

Parameter	Symbol	Definition
Roughness average (dense layer)	$R_{A,\ D}$	$R_{A,D} = \frac{1}{n}\sum_{i=0}^{n}\left\|y_D(i) - y_{D,avg}\right\|$
Total roughness (dense layer)	$R_{T,\ D}$	$R_{T,D} = y_{D,\ max} - y_{D,min}$
Roughness average (roughened layer)	$R_{A,\ R}$	$R_{A,R} = \frac{1}{n}\sum_{i=0}^{n}\left\|y_R(i) - y_{R,avg}\right\|$
Total roughness (roughened layer)	$R_{T,\ R}$	$R_{T,R} = y_{R,\ max} - y_{R,min}$
Roughened layer thickness	T_R	$T_R = y_{F,\ max} - y_{D,avg}$
Hairiness index[a]	H_L	$H_L = 5\left(\frac{L_e}{L_s}\right)$

[a] The coefficient of 5 in the definition of the hairiness index is included for consistency with the standard definition of hairiness index used to measure yarns in the textiles industry, accounting for the fact that our measurements are one-sided (fibers on the underside of the trap are not measured)—so we use a coefficient of 5 rather than 10.

previously developed to simulate the realistic transfer of trace explosives onto a touched object [21]. For this purpose, stock solutions of RDX and TNT in acetonitrile were prepared to 100 μg/mL and 10 μg/mL, respectively, selected based on the instrumental detection limits using the given analytical protocol. Ten 10 μL aliquots of either diluted solution were spiked onto the center of a 5 cm × 2.5 cm coupon of fluorinated ethylene propylene (FEP). The droplets were allowed to dry under ambient conditions until only barely visible explosive residue remained. Immediately after drying, residues were transferred to the substrates, paper or Cordura, by manually wiping the substrate across the FEP. Then, either the TCFG or Nomex trap was manually wiped over the substrate a single time. All swiping was performed by the same researcher with a focus on using consistent speed, hand pressure, and direction of motion. Precision of replicate analyses (five replicates) was monitored to ensure swipe repeatability.

Following transfer of residue, all materials (FEP, substrate, and trap) were placed into separate 4 mL glass vials containing 3 mL of acetonitrile. After a 30 min static extraction, the materials were vortexed for 20 s and the liquid contents were transferred into separate 20 mL scintillation vials. Previous work by DeTata et al. indicated that dissolution into acetonitrile with agitation is sufficient to remove the explosive residues from the materials [22]. The solution was then evaporated under a stream of house nitrogen (8.3 L/min) to dryness (approx. 22 min) leaving behind only explosive residue in the vial. The samples were reconstituted with 2 mL of acetonitrile, vortexed for 20 s, and transferred to 2 mL autosampler vials. At this point, the total explosive material for each sample was divided between the extracts from the FEP, substrate, and trap. Thus, assuming no loss, the maximum concentration of explosives that could be extracted from a single material was 5 μg/mL for RDX and 0.5 μg/mL for TNT. Positive controls were also made to these concentrations.

As RDX is rarely found on its own, C-4 was chosen as a third explosive analyte to represent a more realistic RDX application. Solid C-4 material, having the consistency of a sticky clay, was applied directly to the substrate by wiping the C-4 directly onto the Cordura or paper. The manual swipe and extraction methods were then followed as described above. All C-4 residue was thus divided between the substrate and the trap.

Samples were analyzed by gas chromatography / electron capture detection (GC/ECD) (Agilent Technologies; 7890A GC with μECD) using a method adapted from Field et al. [23]. One microliter of solution was injected into a heated GC inlet (250 °C) at a 10:1 split ratio (chosen for optimal peak shape). The GC column used was an Rtx-5MS (Restek; 15 m × 250 μm × 0.25 μm), and the flow rate through the column was set to 5 mL/min. The column oven was initially held at 100 °C for 30 s, then ramped to 250 °C at a rate of 50 °C/min, and finally held for an additional 2.25 min at 250 °C. The μECD temperature was kept at 275 °C, and the N_2 make-up gas flow rate was 60 mL/min.

It was assumed that there was some analyte loss during the multi-step extraction process. The solvent extraction efficiency was determined by spiking each material directly with 100 μL of the explosives solutions. For the non-absorbent materials (FEP, Cordura, and TCFG), ten 10 μL aliquots were spiked onto the material as done in the dry transfer method. The absorbent materials (Nomex and paper) were spiked while hanging from a thin wire in order to prevent loss of solution due to the liquid soaking through to the underlying surface. The above described extraction method was used, and resulting sample solutions were compared to the positive controls to determine the efficiency of the solvent extraction method. Replicates of ten samples were prepared for each explosive/material combination. The determined extraction efficiency (in percent) was applied to all data to determine the actual amount of explosive residue on each material in a set of samples.

All comparisons were made using a Student's t-test, assuming equal variance, and at a 95% confidence.

3. Results

3.1. Trap surface texture

For the Nomex samples, differences in surface texture across the re-use conditions were readily apparent from a visual comparison of micrographs, as shown in Fig. 3. While the new traps were nearly smooth, the re-used Nomex traps exhibited a roughened layer of loose fibers which generally increased in thickness, fiber number density, and fiber entanglement with the number of swipes. Conversely, in the case of TCFG traps, trap re-use had little effect on texture, as shown in Fig. 4. Unsupported fibers were rarely observed, and there were no apparent changes to the surface after repeated swiping. For both trap types, there were no

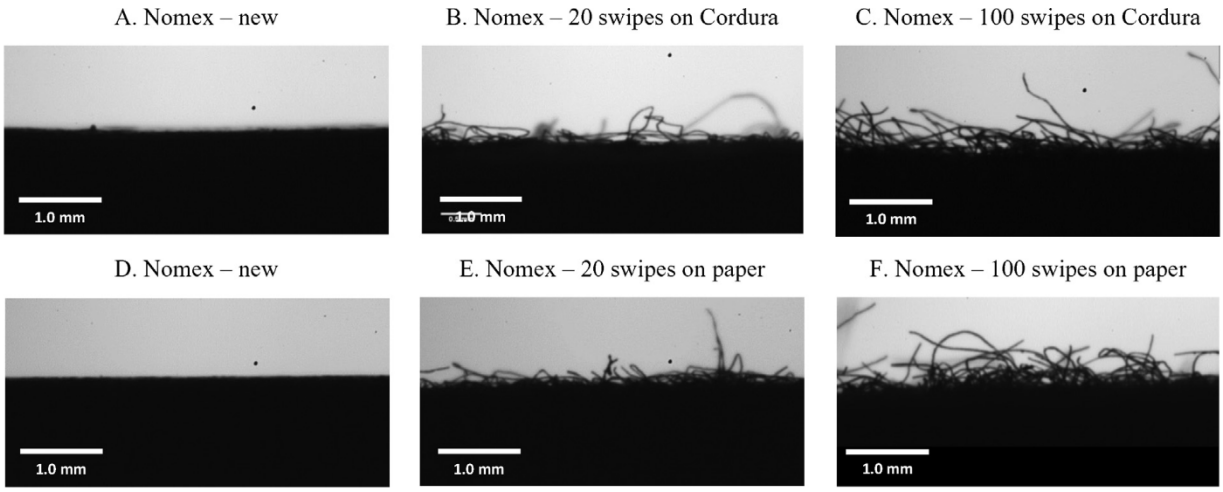

Fig. 3. Examples of Nomex trap surface texture micrographs under different re-use conditions, captured using silhouette microscopy.

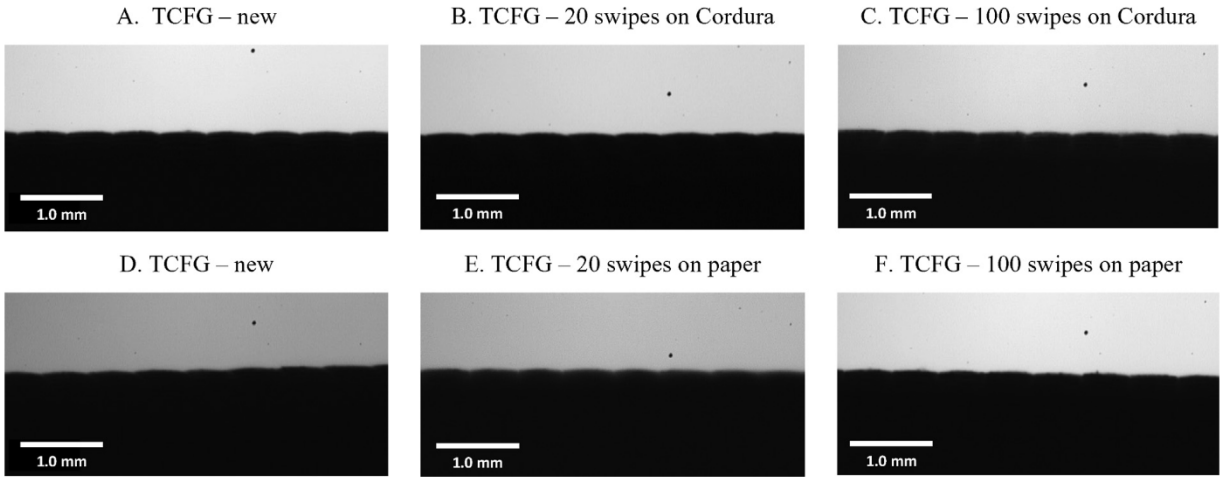

Fig. 4. Examples of TCFG trap surface texture micrographs under different re-use conditions, captured using silhouette microscopy.

obvious differences between the textures of trap samples repeatedly swiped on Cordura versus kraft paper.

Microscopic observations were supported by quantitative results from image processing, as shown in Fig. 5. The same trends were observed across all of the texture parameters. For the Nomex traps, there was a clear relationship between the trap texture parameters and the number of swipes, but the difference between traps worn on different substrates (paper or Cordura) was not statistically significant for any parameter. For the TCFG traps, no statistically significant differences were observed between any of the treatment conditions.

Based on our observations in this study, we consider the hairiness index and roughened layer thickness to be the most important parameters for characterizing the texture of a trap surface, as the combination of these two measurements allows one to distinguish between a smooth surface (low H_L and low T_F), a surface with numerous short fibers (high H_L and low T_F), a surface with a few rogue fibers (low H_L and high T_F), and a surface exhibiting a thick roughened layer of tangled surface fibers (high H_L and high T_F). Such distinctions are not possible from roughness values alone.

A complete table of surface texture results is provided in Supplementary Information.

3.2. Trap efficiency

3.2.1. TNT and RDX

Analyte loss through the extraction process was estimated for each analyte/material combination (for new traps with no roughening). Results are given in Table 2 as percentage analyte recovered compared to the total deposited. Any loss was likely due to analyte left on the trap or substrate materials or the internal surface of the vials, or, to a lesser extent for TNT, due to sublimation. Extraction efficiency was statistically similar (t-test, 95% confidence) for RDX and TNT, with the exception of the Nomex material. In this case, the greater observed loss of TNT may have been caused by retention of TNT by the Nomex material due to forces such as π–π or van der Waals interactions.

Using the dry transfer process, the, analyte (TNT or RDX) was transferred from the FEP to a substrate (Cordura or paper), and swiped with either a TCFG or Nomex trap. The analyte residue on each material following the completion of the dry transfer process was quantified. Examples of the distribution of analyte between each substrate/trap combination are given in Fig. 6, where the amount of analyte recovered from each material is given as a percentage of the total mass of analyte initially loaded onto the FEP (10 μg RDX and 1 μg TNT). On average, approximately 93% (±7%) of

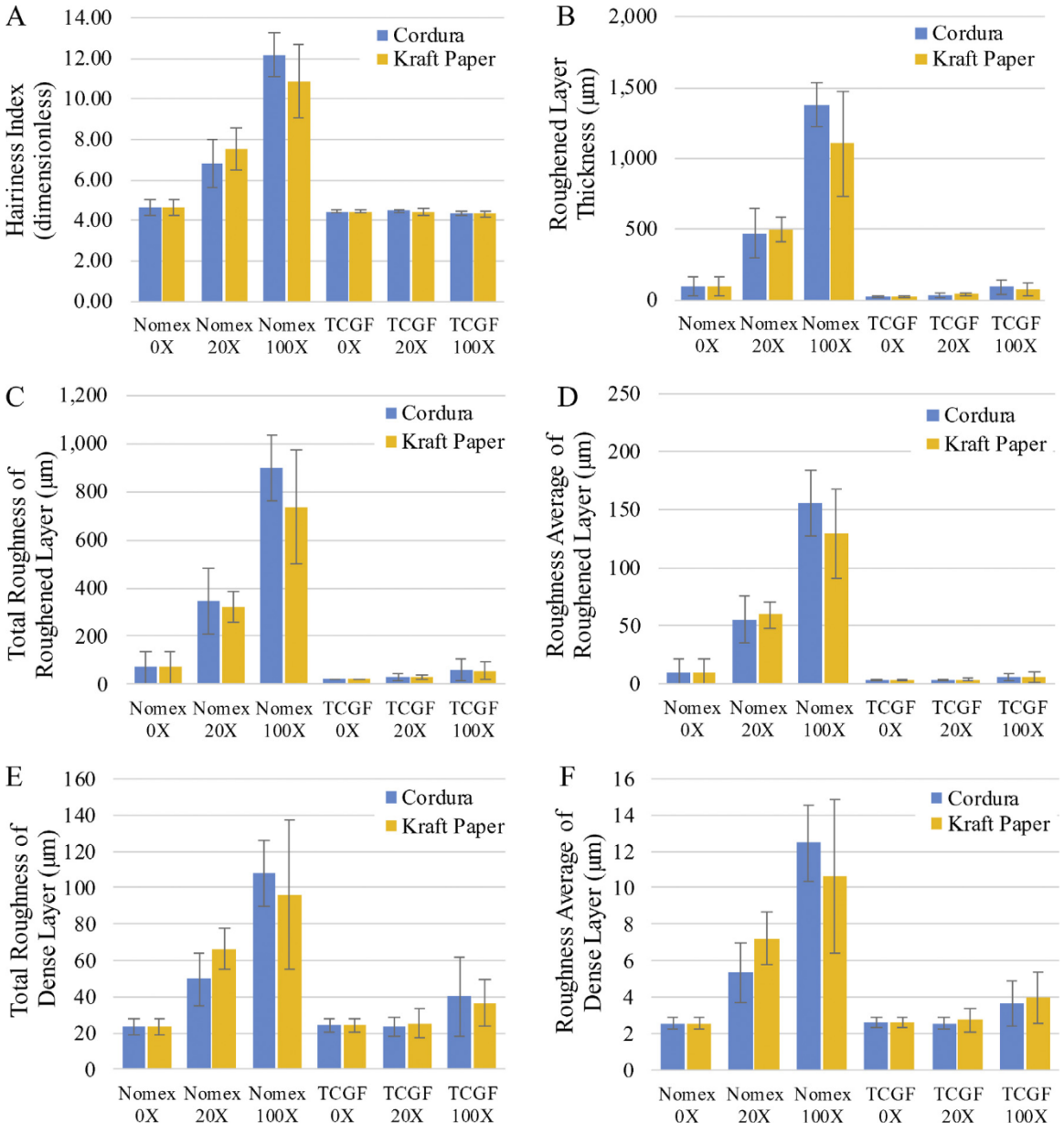

Fig. 5. Surface texture results for Nomex and TCFG traps following zero, 20, and 100 simulated re-uses: (A) hairiness index; (B) roughened layer thickness; (C) total roughness of the roughened layer; (D) roughness average of the roughened layer; (E) total roughness of the dense swab surface; and (F) roughness average of the dense swab surface. All statistics were calculated based on 5 replicates per material/re-use condition and uncertainties represent one standard deviation from the mean.

Table 2
Solvent extraction recoveries of TNT or RDX (given as percentage of total deposited ± one standard deviation) from each material.

Material	TNT	RDX
TCFG trap	94.8% (±6.7%)	104% (±12%)
Nomex trap	77.6% (±3.5%)	91.6% (±6.4%)
Cordura fabric	98.9% (±5.5%)	101% (±15%)
Paper	90.4% (±6.7%)	96.8% (±14.5%)
FEP	109% (±7 %)	103% (±9%)

TNT or RDX was accounted for in the total. The distribution of TNT was similar between all substrate/trap combinations with approximately half of the TNT being left on the substrate (51% ± 11%), and one third (37% ± 6%) left on the FEP (Fig. 6A). The amount collected by the trap varied from less than 2% to nearly 10% depending on trap material and re-use. The distribution of RDX was more variable with substrate and trap material (Fig. 6B) than TNT. With RDX, significantly more residue was transferred from the FEP to the substrate (t-test, 95% confidence), with an average of only 7.5% (±6.3%) of the total RDX left on the FEP. The amount transferred from the substrate to the trap varied from 1.4% up to 57% for the highly worn Nomex.

In comparisons of collection efficiency (irrelevant of the transfer efficiency of material from the FEP to the substrate), the amount of analyte recovered from trap and the substrate were totaled, and data are listed as the percentage of this total recovered from the trap (i.e. trap recovery efficiency = mass on trap / [mass on substrate + mass on trap]). Fig. 7 compares trap collection efficiency of each analyte from both substrates across three use levels of the Nomex and TCFG traps.

The most obvious trend is that the Nomex out-performed the TCFG in all scenarios, with a collection efficiency twice to more

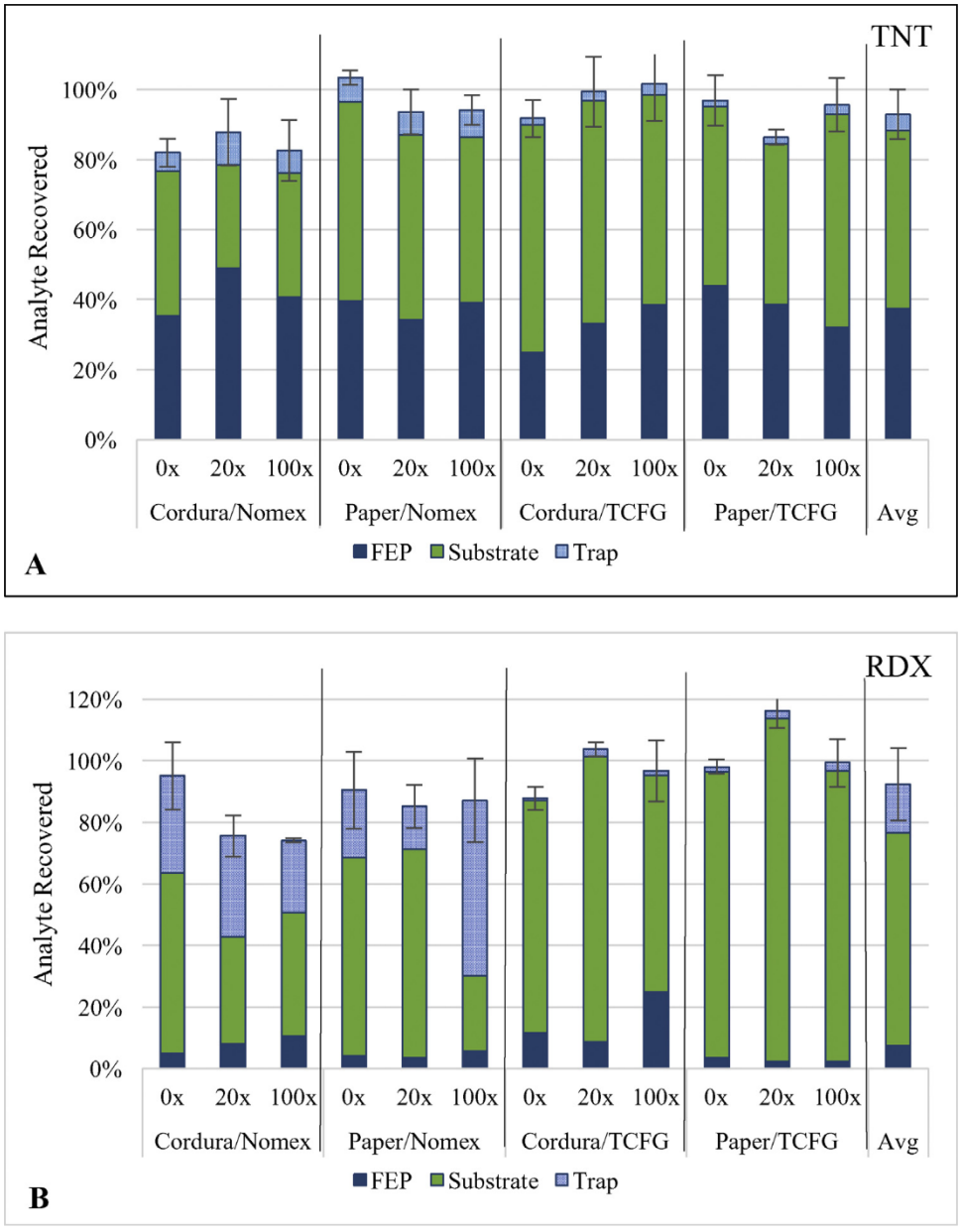

Fig. 6. Averaged distribution of analyte, (A) TNT and (B) RDX, across FEP, substrate (Cordura or paper), and trap (Nomex or TCFG) using dry transfer method. Data given as percentage of total analyte initially deposited onto FEP, and error bars equal one standard deviation of the averaged total amount of analyte for each data point.

than ten times greater than the TCFG in all substrate/trap combinations.

It was hypothesized that re-use of the trap materials would affect collection efficiency by roughening the surface. For the TCFG traps (Fig. 7 A–D), collection efficiency did appear to increase slightly with trap wear, although such changes were minimal and not statistically significant (t-test, 95% confidence). There was no statistically significant difference between the collection of RDX and TNT, although the collection of TNT was generally higher.

For the Nomex collection of both RDX and TNT from Cordura (Fig. 7A and C), the medium wear (20×) had the highest collection efficiency, and this was statistically higher than the other use levels (t-test, 95% confidence), excluding RDX with the 100× swipe. Nomex collection from paper (Fig. 7B and D), on the other hand, showed an increased collection efficiency after 100 repetitive swipes for both analytes, though this difference was particularly drastic for RDX residue (Fig. 7D). The differing results between the two substrates could be related to how each substrate physically interacts with the Nomex traps. The swiping of each trap was done on the same substrate it then sampled; i.e. traps that were used with Cordura were also repetitively swiped (20 or 100 times) on Cordura, and the same with the paper. This would result in traps with different wear patterns between those traps worn on Cordura vs. paper, likely affecting collection efficiencies with increasing deterioration; however, these differences were not quantified in the surface texture measurements (Fig. 5). Previous publications have suggested that the removal of explosive particles from a substrate is more affected by the substrate texture and explosive particle shape, than by interactions with their composition, although both do play a role [6,7]. Cordura is a nylon-based material with tight 6-carbon chains, while paper is comprised of cellulose, with a backbone containing many hydroxyl groups, and thus differing van der Waals and H-bonding interactions may play a role as well.

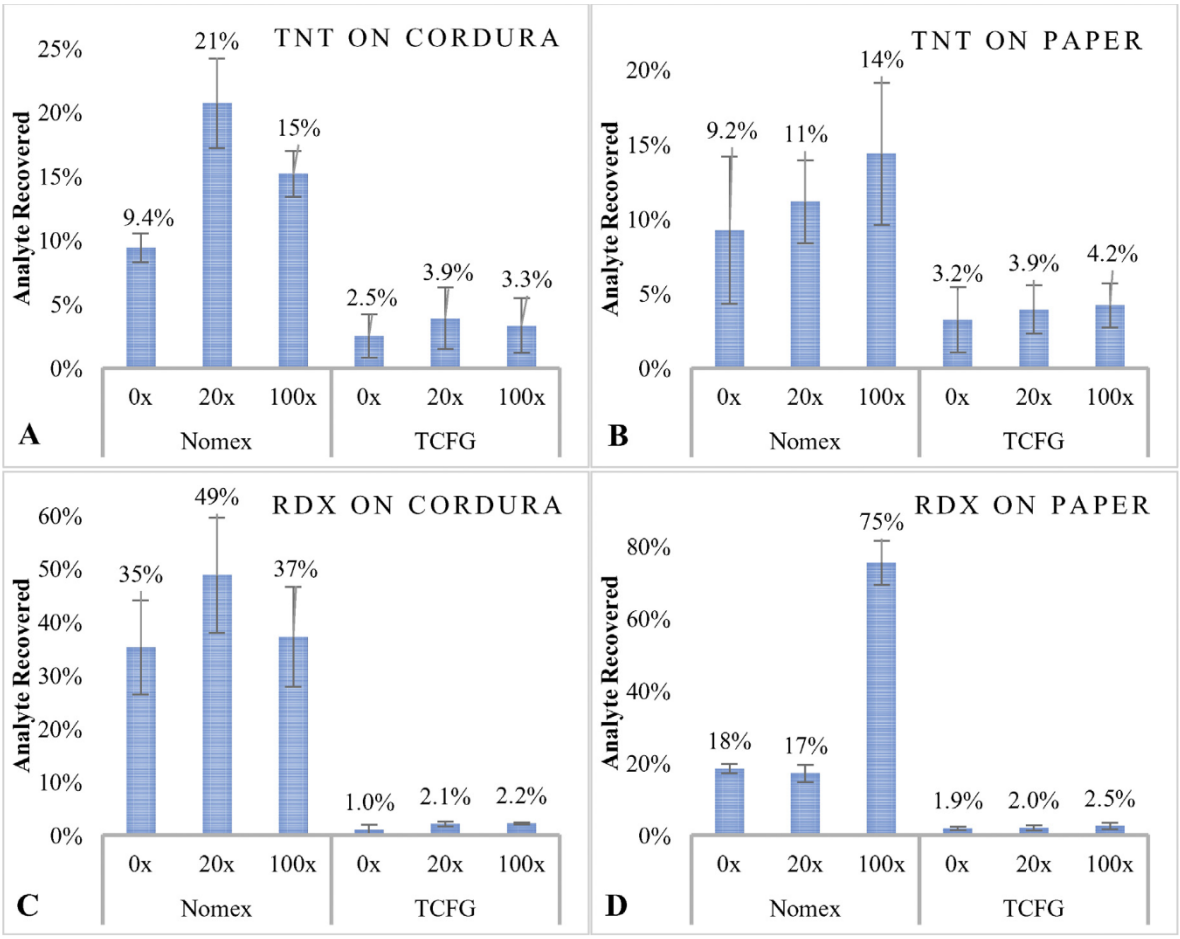

Fig. 7. Collection efficiency of Nomex traps and TCFG traps of (A) TNT from Cordura fabric, (B) TNT from paper, (C) RDX from Cordura fabric, and (D) RDX from paper. Re-use of the Nomex and TCFG traps are compared following no wear (0×), medium wear (20×), and high wear (100×).

3.2.2. Composition 4 (C-4)

Solid C-4 was directly applied to the substrate of choice, and thus the percentage given in Fig. 8 was determined as mass on trap divided by total mass on trap and substrate only. Data for RDX transfer from Cordura and paper are given in Fig. 8. The most notable result is the comparably poor recovery of C-4 by both traps (all use levels) from the paper substrate (Fig. 8B) though the Nomex trap performed marginally better than the TCFG (only 20× statistically significant; 95% confidence). It is likely that the plasticizers in C-4 have greater affinities for paper than for either trap, thus impeding collection. Collection of C-4 from the Cordura was also relatively poor for the TCFG trap (Fig. 8A) and worsened with increased wear. The greatest recovery of the RDX from C-4 residue was by the Nomex trap from Cordura (Fig. 8A) with

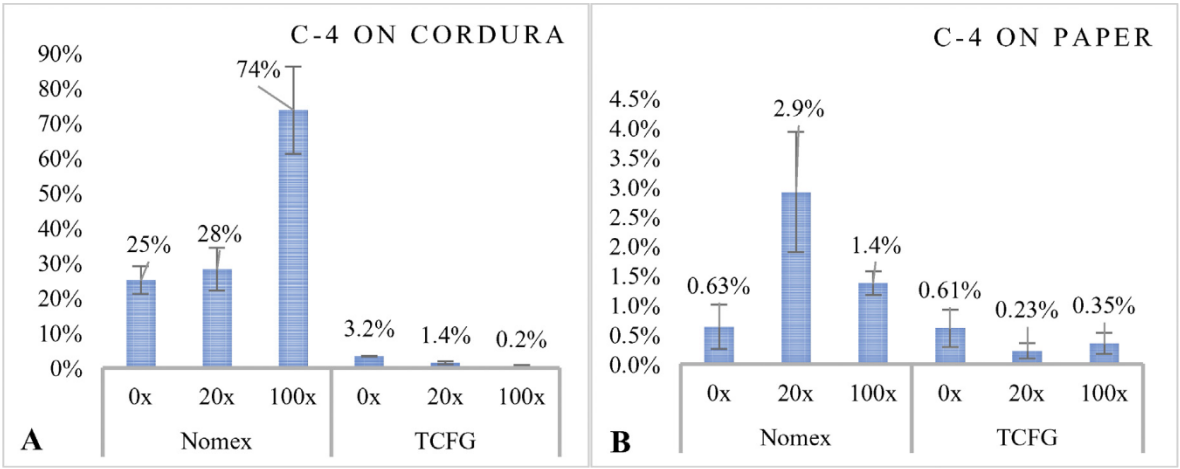

Fig. 8. Collection efficiency of Nomex and TCFG traps of C4 from (A) Cordura fabric, (B) paper, given as percentage of total RDX deposited. Wear levels of the Nomex and TCFG traps are compared following no wear (0×), medium wear (20×), and high wear (100×).

collection efficiency statistically similar to RDX from paper. Furthermore, recovery from the highly worn (100×) trap was significantly greater than from the lesser used materials.

4. Discussion

This study investigated the effects of trap re-use on both the trap's physical surface texture and its effectiveness in collecting explosives residues. Nomex traps were readily roughened by repeated swiping, generating a roughened layer of looped and protruding fibers on the trap surface, whereas repeated swiping produced little to no effect on the TCFG traps. Silhouette microscopy was used to observe and quantify the level of trap roughening. In particular, two new parameters were used to characterize the traps' surfaces: the roughened layer thickness (T_R), which quantifies the maximum extension of a loosened fiber from the surface; and the trap hairiness index (H_L), which quantifies the number and length density of such fibers. These parameters were highly correlated with the trap re-use condition. Roughness values (R) generally followed the same trends; however, roughness is not considered ideal as an assessment metric due to potentially confounding effects of long fibers or thick fuzz on many of the trap samples.

In general, measured collection efficiencies do correlate with surface texture measurements: Nomex collection efficiency increased, as did its roughness, with repeated swiping. Also, TCFG exhibited little roughening with re-use, and at the same time there was little improvement in collection efficiency. However, the measured collection efficiencies with increasing trap use were found to be more nuanced than surface texture measurements. For example, there was no statistical difference (t-test, 95% confidence) in the roughening by Cordura versus paper, but collection efficiencies did indeed differ between the two. This implies that substrate / trap / analyte interactions are complex and affected by a variety of factors that are not fully represented in the surface texture measurements.

Comparing morphology changes noted by SEM imaging and measured collection efficiency changes as reported in Fisher et al. [10] and Staymates et al. [11] to the work herein also implies a complex relationship between trap surface and collection efficiency. For the Nomex traps, Fisher et al. observed loosening of the trap weave and increased porosity with continued re-use. This is consistent with the surface texture measurements made in this work. On the other hand, Staymates did not find any notable change in surface texture or weave with increased wear. These differences are interesting as it was this (NRL) work and Staymates et al. that used similar substrates and methodologies for swiping, while Fisher et al.'s study involved roughening by repeated swiping of airport test surfaces with inherent greater variation than found in the other studies. The notable differences between this (NRL) and Staymates et al. swiping methodologies was the speed of the mechanical swipers, 30 mm/s and 70 mm/s (as stated in [5]), respectively, and the use of automated versus manual swiping for particle collection. Fisher et al. did not list swiping speed, as this and other factors such as pressure, swipe area, etc. likely varied significantly between airport personnel. It is possible that differences in swiping speed or the use of automated versus manual sampling could account for these differences.

In contrast, comparing collection efficiency data for Nomex from each research group (Fig. 9), NRL and Staymates et al. yielded similar aggregated results with increased sampling efficiency for increased wear, although NRL used actual explosive residue and

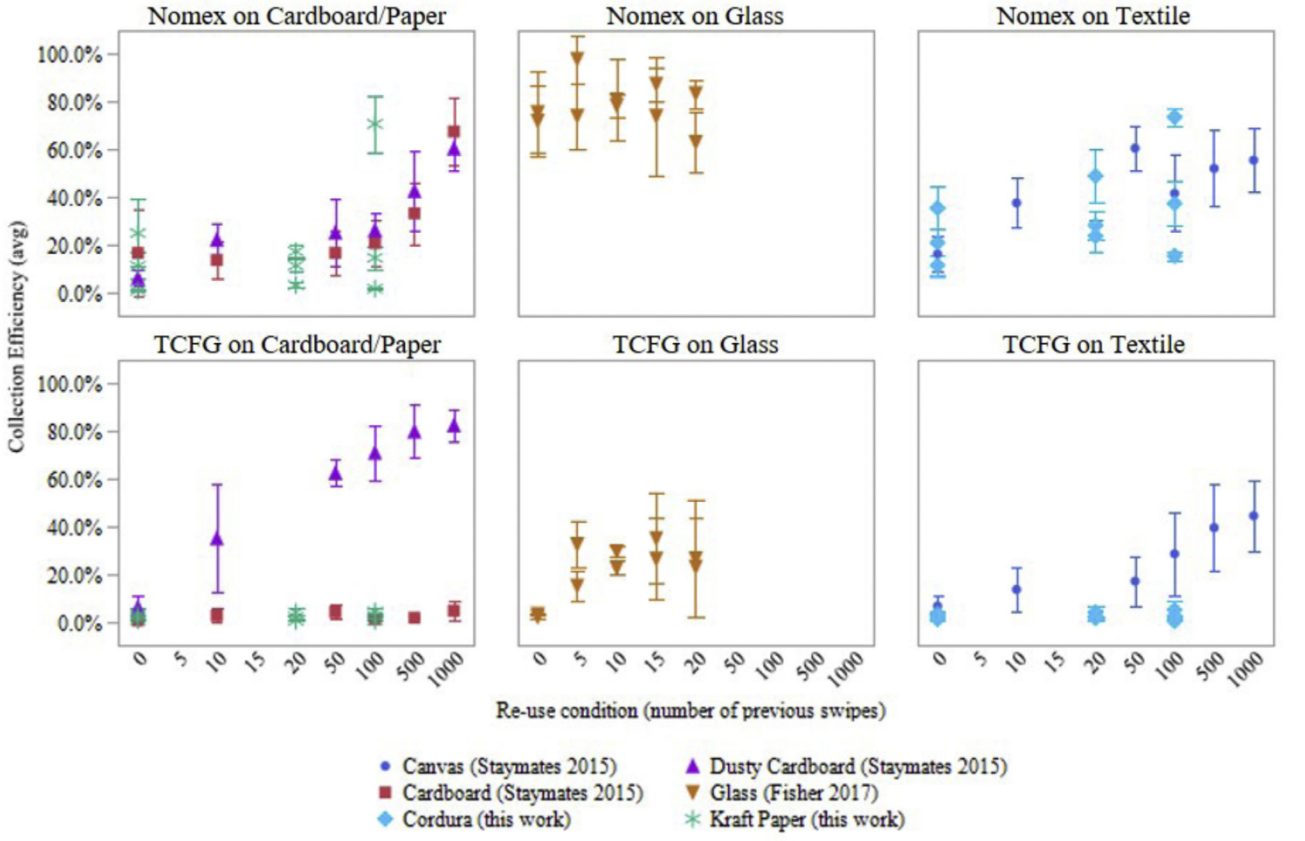

Fig. 9. Collection efficiency vs. number of previous swipes for Nomex and TCFG traps, grouped by the type of substrate, based on data aggregated across three studies (this work, Refs. [10] and [11]). NRL TNT and RDX data was aggregated for this figure.

Staymates et al. used polymer microspheres. Re-use on the cardboard substrate (similar to NRL's paper substrate) increased collection efficiency out to 1000 uses in Staymates et al. NRL saw a similar efficiency for TNT and RDX, but not C-4 whose collection efficiency was much lower and peaked at 20 uses. On canvas (similar to NRL's Cordura substrate), Staymates et al. saw improved collection efficiency to 50 uses. This is similar to NRL's data which showed peak efficiency at 20 uses for TNT and RDX (but not C-4). This seems to suggest that when substrates are similar, polymer microspheres are good surrogates for TNT and RDX particles for Nomex trap swiping, but as dry beads they may not be representative C-4 residues that include binders. More research should be done to further understanding of these disparities.

Fisher et al. sampled RDX and HMX residues from glass and achieved significantly higher efficiencies than the other two studies. Collection efficiencies for both traps (Fig. 9) from the glass were comparable to the collection efficiencies NRL measured for RDX from Cordura (Fig. 7). This might indicate that Cordura (non-porous) behaved more similarly as a substrate to glass than the paper (porous). The similarity between this study and Staymates et al. and dissimilarity to Fisher et al. appear to be due to the differences in interactions between analytes and swiped substrate (i.e. paper / canvas versus glass) more so than the morphology changes related to the type of substrate used for swiping (i.e. repeated swiping of airport test surfaces versus clean canvas/Cordura or paper/cardboard).

Results from NRL's surface texture measurements and Fisher's SEM imaging of the Nomex wipes yielded similar findings, though these differed from imaging by Staymates et al. Conversely, for the TCFG traps Staymates et al. and NRL saw minimal to no changes or signs of degradation in the TCFG traps with re-use (with the exception of the dusty cardboard), while Fisher et al. did note fiber ruptures and increased surface roughness with subsequent swipes. This increased roughening could be caused by dust/debris on the airport test surfaces sampled in that study. However, again, the collection efficiency data did not necessarily correlate with the morphology changes. Overall Fisher et al. and Staymates et al. saw significantly higher collection efficiencies than NRL (Fig. 9) for TCFG traps. All three research studies showed poor collection (6% or less) from fresh TCFG traps, but NRL recorded no or minimal improvement in collection efficiency with increased usage of the traps; the average collection efficiencies for TNT, RDX, and C-4 were only 3.0%, 1.9%, and 1.0%, respectively, with little effect from the type of substrate (Figs. 7 and 8). The collection efficiency data from Staymates et al. for TCFG from clean cardboard was similar to NRL, but their collection from dusty cardboard and canvas each exhibited a collection efficiency that increased with the number of swipes. Staymates et al. suggest that this increase is due to the wearing down of the Teflon coating, which is supported by their SEM imaging of wear by dusty cardboard (though not by their SEM imaging of wear by canvas). Alternatively, they suggest that the increase is due to an increased electrostatic effect, which could have a greater effect on the polymer microspheres than actual explosives. Other factors that might contribute to discrepancies between NRL and other research include particle size and geometry (e.g., the use of microspheres vs. non-uniform explosives residues), differences in automated mechanical versus manual swiping, or differences between sampling and /or substrate materials.

5. Conclusion

This work studied the effect of trap wear (re-use) on collection efficiency and, for the first time, was correlated to quantifiable morphology changes evaluated using a silhouette microscopy technique developed for the measurement of material texture. Two new texture parameters (hairiness index and roughened layer thickness) were defined and used in combination with standard measures of surface roughness to quantify various attributes of trap surface texture. We demonstrate that Nomex trap surface texture changes significantly with repeated re-use, including liberation of fibers from the nonwoven matrix and thickening of a "roughened layer" of loose surface fibers. The degree of surface roughening is directly related to the number of times the trap has been re-used. In general, Nomex trap collection efficiency also increased with increased wear. Meanwhile, Teflon-coated fiberglass (TCFG) traps showed little textural change with repeated swiping and minimal to no improvement in TCFG trap collection efficiency. Comparing the aggregated data from our study with those of other researchers show a basic agreement that increased trap wear tends to correlate with increased roughness and with increased collection efficiencies. However, collection efficiency is influenced by a variety of factors simultaneously making it difficult to delineate specific trends. Differences in trap, analyte, and substrate properties, including morphology (e.g., hairiness, roughness, and porosity), surface interactions, and adhesion forces can all impact collection efficiencies. Differences between collection efficiencies for RDX particles deposited from solution versus more realistic C-4 also emphasize the importance of considering field-relevant materials in future studies. Based on this work, further validations would be recommended to develop new protocols using pre-roughened traps. Alternatively, development of novel trap materials with a rougher surface might enhance trace explosive residue collection.

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