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Supercritical Fluid Cleaning of Banknotes

Nabil M. Lawandy*,[†,‡] and Andrei Y. Smuk[†]

[†]Spectra Systems Corporation, Providence, Rhode Island 02903, United States

[‡]School of Engineering and Department of Physics, Brown University, Providence, Rhode Island 02906, United States

ABSTRACT: With nearly 150 billion new banknotes being manufactured and printed every year around the world, the replacing of unfit currency is approaching \$10 billion annually. In addition, central banks must also deal with the environmental challenge of annually disposing of nearly 150,000 tons worth of notes unfit for recirculation. Seminal work by the De Nederlandsche Bank (DNB) has identified that soiling is primarily a yellowing of the notes due to the accumulation of oxidized sebum. We show that supercritical CO_2 (SCCO2) can be effectively utilized to remove sebum and other oils and contaminants, including common bacterial colonies, from both paper and polymer banknotes without destroying the costly and sophisticated security features employed by central banks to prevent counterfeiting. SCCO2 cleaning at 60°C and 5000 psi was shown to be effective in cleaning conventional straps of 100 banknotes, extracting nearly 4% of the initial strap weight. Measurements of note soiling distributions on a banknote sorting machine running at 10 banknotes per second showed a significant shift in soiling levels after cleaning, supporting the claim that processing of SCCO2-cleaned notes would result in significantly fewer notes being classified as unfit due to soiling and shredded.

■ INTRODUCTION

Central banks around the world, from the Federal Reserve Bank of the United States to the Reserve Bank of India, sort banknotes using high speed machines to interrogate them for authenticity and for fitness. The largest sorters operate at 40 banknotes per second and can have as many as 16 sensors to detect counterfeits and notes which are not fit for recirculation.[1] The fitness sensors operate primarily on optical image analysis to detect tears, tapes, graffiti, and soiling. Additional sensors are often used to determine banknote limpness as another metric for whether the notes are fit or have to be replaced. The most common cause leading to a banknote being rejected is soiling.

Work in the past decade has shown that the primary source of soiling is the transfer of human sebum to the banknote surfaces and its subsequent oxidation that results in a yellowish product that absorbs in the visible region of the spectrum.[2−4] The transfer of sebum to banknotes results in changes in color, and in uncoated notes, there is also the possibility of a change in scattering due to the index matching created by the infiltration of the sebum material into a cellulosic network. This index-matching effect is absent in polymeric banknotes and banknotes with a protective coating that have essentially no porosity. Both of these effects result in a soiling that leads to rejection of the banknotes by the sensors in the processing machines. According to data from the De Nederlandsche Bank, sensors reject 60−80% of notes processed around the world on the basis of soiling.[5]

Banknotes that are rejected by the fitness sensors are shredded and replaced by new banknotes. In the United States, this results in the placement of an order by the Federal Reserve Bank of the United States to the Department of the Treasury and the Bureau of Engraving and Printing to produce replacement notes. The replacement orders can range from 7 to 11 billion banknotes annually in the United States and is approaching 150 billion worldwide, with much of the growth in volume over the past decade coming from the Peoples Republic of China. The average cost per 1000 banknotes, averaged over all denominations, is estimated to be \$65.[6] The high denomination banknotes have several public features as well as covert machine-readable security features that significantly increase their cost. For example, in the United States, the \$1 denomination costs \$0.055/note, while the new \$100 denomination banknotes to be released in October 2013 will cost \$0.126/note.[7]

The combination of the replacement volumes and the averaged worldwide cost per note results in governments spending nearly \$10 billion annually to provide the public with fit banknotes.

In addition to the cost of replacing banknotes that are not fit, the central banks have to dispose of nearly 150,000 tons of

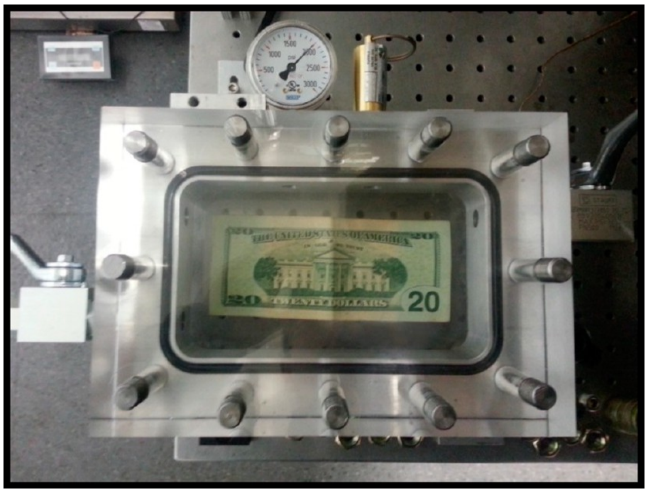

Figure 1. High pressure CO_2 cell in the supercritical phase (60 °C, 2000 psi).

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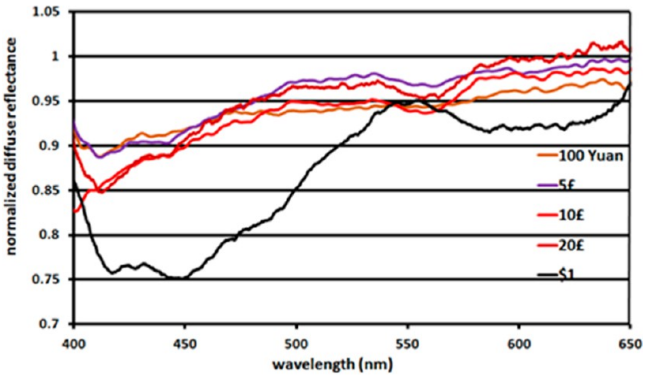


Figure 2. Diffuse reflection spectrum of a number of world banknotes before any application of sebum. The diffuse reflectance was normalized to a white integrating sphere standard.

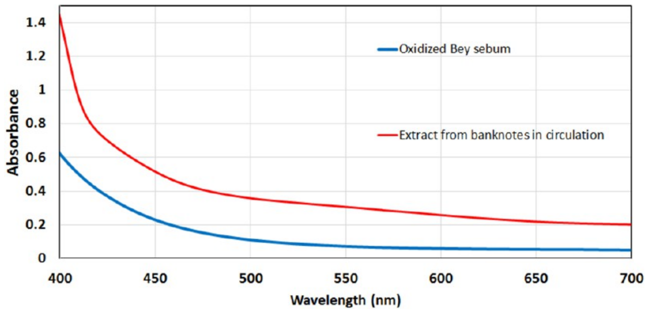


Figure 3. Absorbance spectra of 1 mm thick samples of oxidized Bey sebum and the material extracted from circulation banknotes.

shredded banknotes annually. Although this waste does not contain hazardous levels of toxic materials, it still poses a significant environmental concern.

Supercritical fluids such as CO_2 have been used for specific molecular extraction as well as cleaning operations.[8] The solvating power of supercritical fluids has been shown to exhibit an exponential increase with small changes in temperature and pressure far exceeding what would be expected from solubility calculations.[9] CO_2 has been the most widely used molecule for these applications with a critical point at 72.9 atm and 304.25 K.[10] The supercritical state, which exhibits a density near that of a liquid but has the space filling properties of a gas, has been effectively used to extract certain molecules for biotechnology applications, to remove grease and other substances from high performance parts, and as an alternative to dry cleaning.[11,12] In addition, it has been demonstrated that supercritical fluid mixtures are effective in removing fingerprints from various surfaces.[13−15]

In this work, the use of supercritical CO_2 for cleaning banknotes is shown to be effective in the removal of oxidized

Figure 5. Comparison of a United States $1 banknote before and after coating with an oxidized sebum layer. The top half of the note is coated with sebum and oxidized.

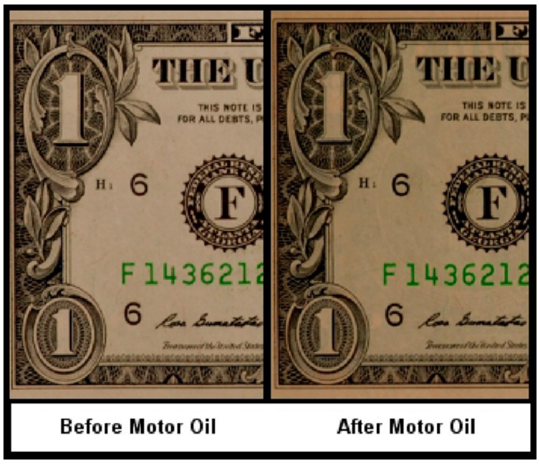


Figure 6. Side-by-side comparison of an uncirculated United States banknote and one stained with motor oil.

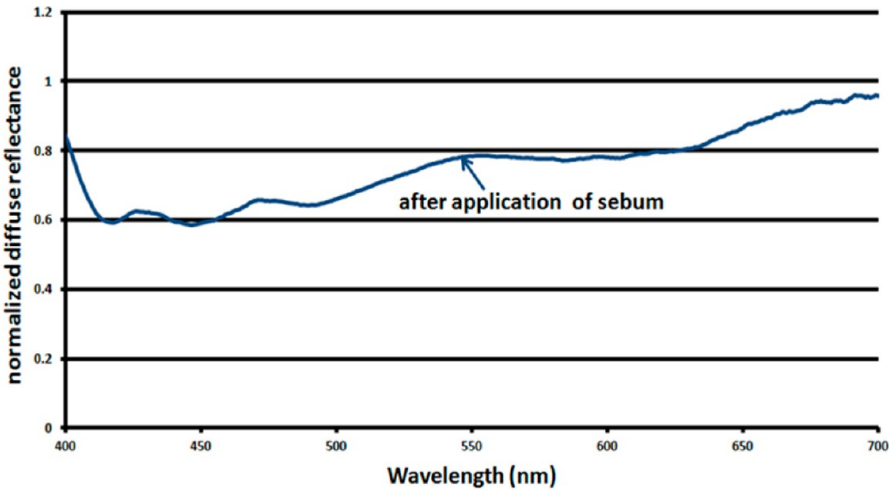


Figure 4. Diffuse reflection spectrum of a sebum-treated United States $1 banknote normalized to a white integrating sphere standard.

Figure 7. Banknotes before sebum treatment, after sebum treatment, and after cleaning with supercritical CO_2 at 60 °C with ultrasonic agitation.

sebum, while leaving expensive security features such as holograms, optically variable inks, security threads, phosphorescent inks, and microprinting intact. In addition, we show that machine-readable features such as magnetic signatures used in single note acceptors such as change and vending machines also remain functional after cleaning.

■ EXPERIMENTAL APPARATUS

The experiments were undertaken using CO_2 as a supercritical fluid in the cell shown in Figure 1, with top and bottom windows for when optical observation was required during the supercritical phase. The supercritical pressure vessel was constructed of a frame machined from a single billet of 6061-T6 aluminum with a top and bottom plate constructed of 2″ thick clear cast acrylic. A quantity of 12 1/2″-13 Grade 8 threaded rods were threaded into the frame and 24 1/2″-13 Grade 8 nuts and 24 18-8 stainless steel flat washers were used to secure the top and bottom plates. An O-ring channel was milled into the frame at the interface of both the top and bottom plates for installation of an ethylene propylene diene monomer (EPDM) rubber O-ring of standard size AS568A-365 to form the chamber seal. The pressure vessel had threaded fittings machined through the frame wall into the cleaning chamber for filling and purging and a second set of threaded fittings for installation of a pressure monitor and a safety release valve. Using an externally wrapped thermal heating tape, the cell could be operated in the range of temperatures from 25−60 °C and at pressures up to 2000 psi. In addition, the cell was immersed in an ultrasonic bath to enhance the cleaning process.

A second generation cleaning cell was a cylindrical 1 L vessel made from a hardened 17-4 grade stainless steel, with the pressure limit of 10,000 psi. It was equipped with additional inlet and outlet ports and a liquid CO_2 pump to allow CO_2 flow though the cell when the latter was pressurized. Solubilized extract was thus continuously carried out of the cell, and after CO_2 pressure reduction across a micrometering valve to the atmospheric value, arrived into a glass vial and precipitated in it, while CO_2 was disposed. During cleaning experiments, the flow of CO_2 at atmospheric pressure was set at 10 L per minute. There was a heating jacket around the vessel. The volume of the vessel accommodated two 100 banknote straps at a time. The possibility of cleaning banknotes in straps is an important practical aspect of handling a large number of banknotes because it is standard for central banks.

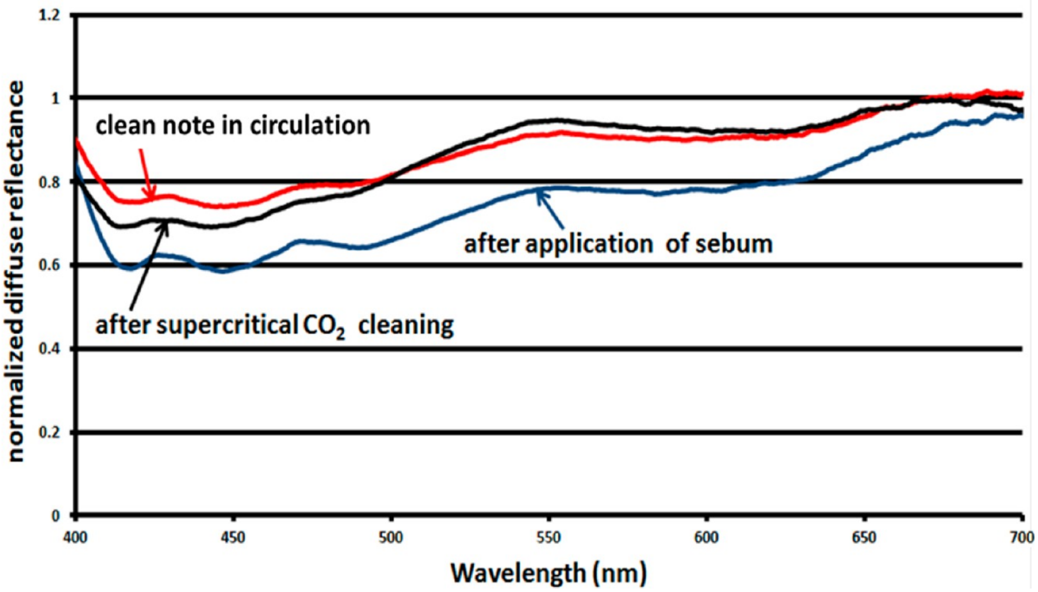


Figure 8. Diffuse reflection spectra for the note before and after sebum application and after supercritical CO_2 cleaning.

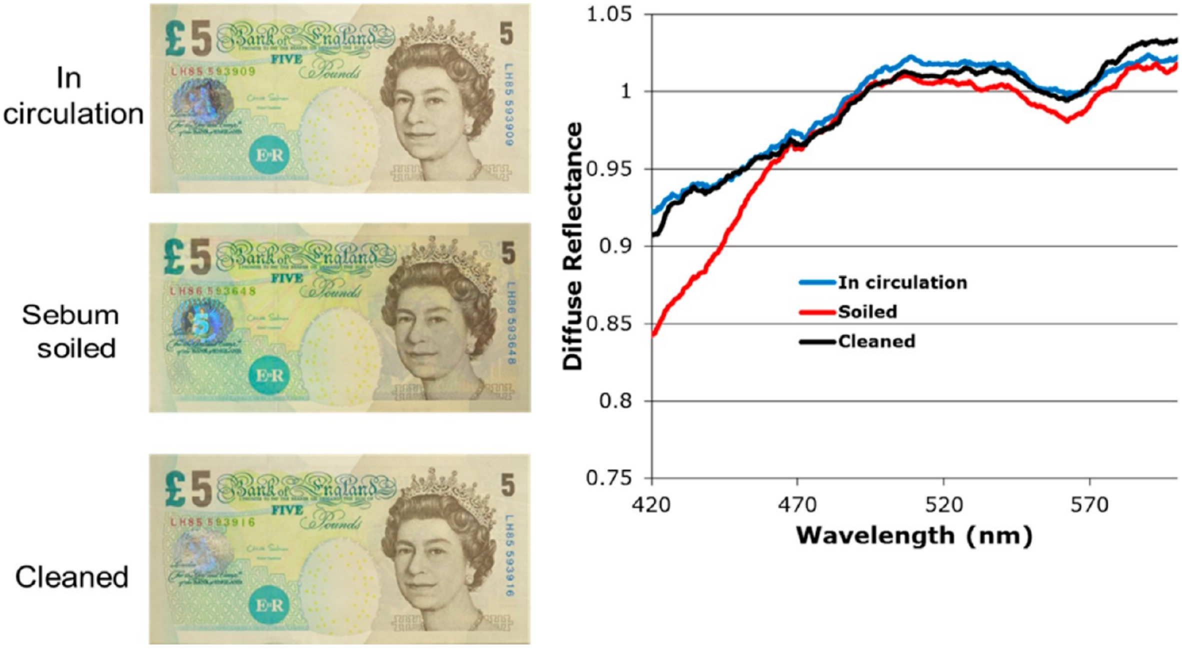


Figure 9. Banknote images and diffuse reflectance data of £5 banknotes treated with sebum and SCCO2 cleaned.

SAMPLE PREPARATION

Experiments were performed using primarily uncirculated banknotes which were "soiled" using the procedure established by the De Nederlandsche Bank.[2] An examination of banknotes from around the world reveals that there is a large variation in the diffuse reflectance spectrum of the uncirculated banknotes shown in Figure 2, with some denominations of Unitd States banknotes having a yellowish tint when manufactured, depending on the specific series. Other banknotes such as the euro, British pound, and the yuan have a much less tinted appearance. In all cases, the substrates used for banknotes do not contain optical brighteners such as stilbenes and are considered to be UV dull.

The process of simulating circulation soiling uses Bey sebum, which is composed of beef tallow (32.8%), free fatty acids (18.0%), lanoline (18.3%), fatty acid tryglycerides (3.6%), cholesterol (3.7%), hydrocarbon mixture (12.0%), and cutina (11.6%). The banknotes were coated using a blade and then oxidized for 8 days under conditions of 65% humidity and 90 °C. Immediately after the banknotes are coated with the Bey sebum and prior to oxidative treatment, heavily coated notes appear translucent as the sebum creates an index matching with the cotton-based substrates. After oxidation takes place, the sebum develops a yellowish color, which along with the index matching effects results in a soiled note very much resembling what is found in circulating currency. Measurements on the melting temperature of the Bey sebum before oxidation gave a value of 42 °C and a value of 36 °C after oxidation. A spectral analysis of the oxidized Bey sebum comparing it to the material extracted from banknotes in circulation is shown in Figure 3. These spectra were taken for 1 mm thick samples and support the modeling of notes soiled in circulation using oxidized Bey sebum. Although the magnitudes of absolute absorption coefficients are different, the spectral signatures are very similar.

Figures 4 and 5 show the diffuse reflectance measurements on a $1 note and the note before and after application and oxidation treatment with Bey sebum, respectively.

Other common soiling mechanisms include coffee and oil stains that occur in normal use of currency in circulation.

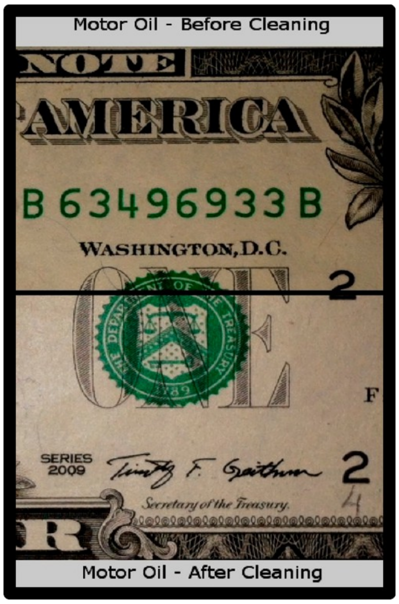


Figure 10. United States $1 banknote soiled with motor oil before (top) and after (bottom) cleaning.

Figure 6 shows a $1 banknote before and after staining with motor oil.

SUPERCRITICAL FLUID CO$_2$ CLEANING EXPERIMENTS ON LABORATORY-SOILED BANKNOTES

Banknotes that were prepared using the coating and oxidation of sebum and staining with motor oil were characterized before and after the supercritical cleaning process, which was comprised of 1−3 h long treatments in the chamber described at temperatures of 60 °C and pressures of 1600−2000 psi.

Characterization was aimed at determining the degree to which sebum was removed and the survivability of various security features. These include ambient light features, security features

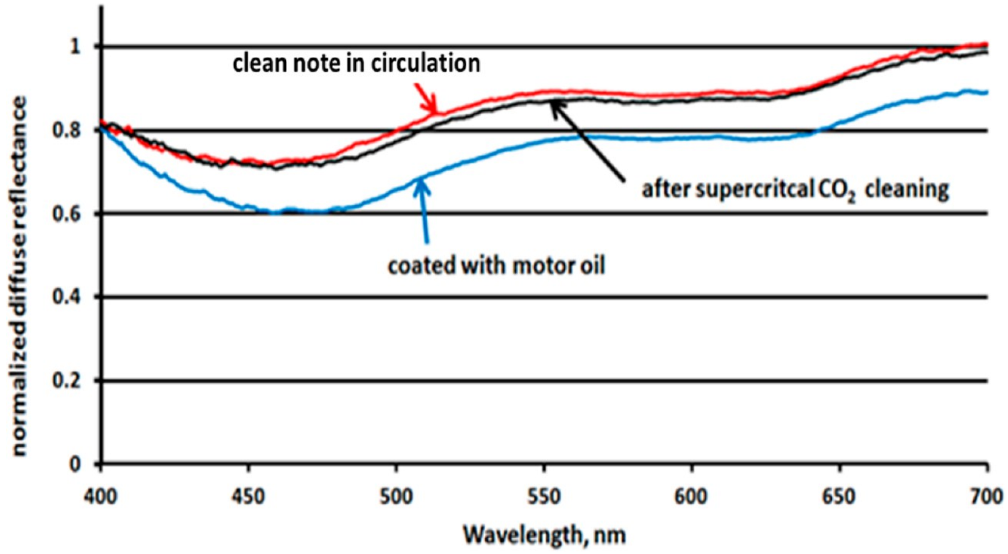


Figure 11. Diffuse reflectance spectra before and after cleaning the banknote soiled with motor oil.

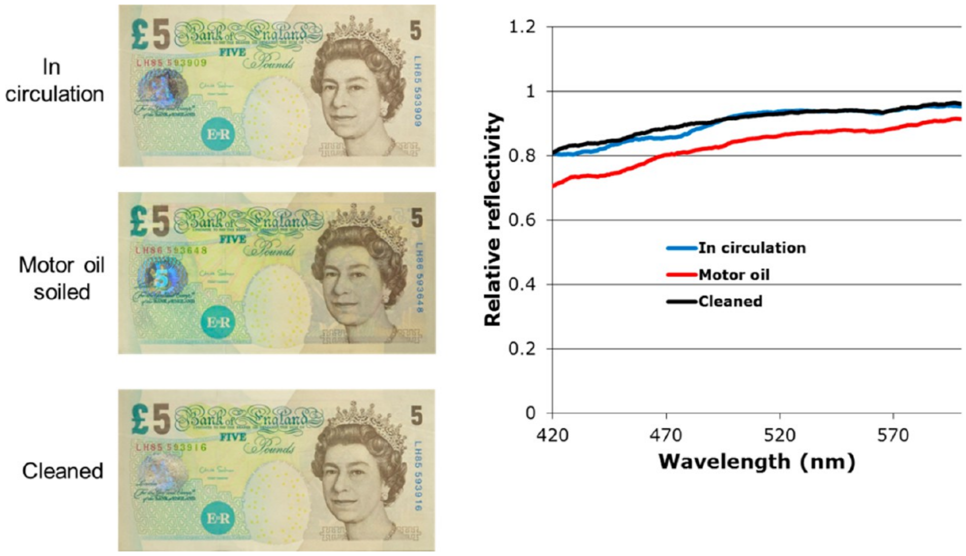


Figure 12. Banknote images and diffuse reflectance data of £5 banknotes treated with motor oil and SCCO2 cleaned.

viewed under UV light, machine-readable capacitive and magnetic features, and high-level covert central bank features such as Enigma (De La Rue plc) and M-Feature (Giesecke and Devrient GmbH). The removal of sebum was studied by measuring the diffuse reflectance spectrum. UV features were characterized before and after using a Horiba Raman calibrated fluorimeter.

The primary goal of the use of SCCO2 cleaning was to assess the removal of oxidized sebum from soiled banknotes and to determine how printing and security features are affected by the process. Experiments were performed on a number of banknotes with a focus on United States banknotes made from paper, which is approximately 75% cotton and 25% linen fibers and printed by the United States Bureau of Engraving and Printing. The diffuse reflectance spectrum of the sebum-coated banknotes was measured before and after cleaning using a broadband LED and an Ocean Optics spectrometer. Figure 7 shows images of a $1 note before and after application of sebum and after cleaning in SCCO2.

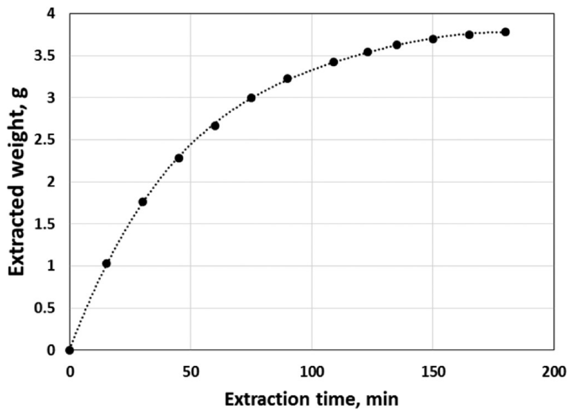


Figure 13. Weight of material extracted from a strap of 100 ($5) banknotes as a function of extraction time.

The diffuse reflectance measurements shown in Figure 8, which mirror the changes in the images in Figure 7, quantitatively show that the cleaning process was effective in removing the applied oxidized sebum layer. In addition, closer examination of the banknote with oxidized sebum shows that the sebum penetrated into the note and produced an index of refraction

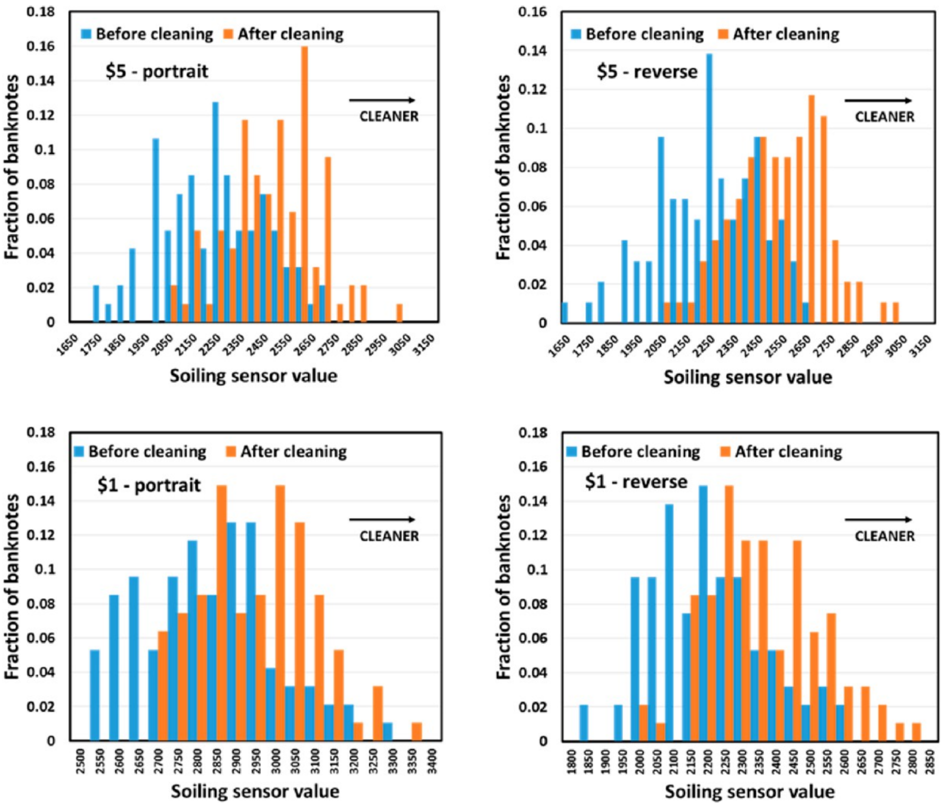


Figure 14. Soiling-level distribution before and after supercritical CO_2 cleaning of \$5 and \$1 straps containing 100 banknotes each.

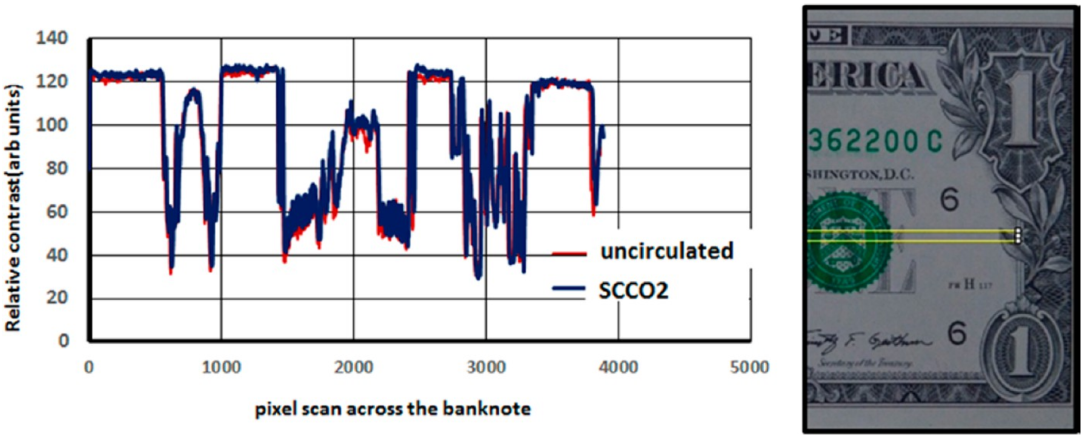


Figure 15. Unaltered pixel contrast across a scan of a banknote before and after the cleaning process is shown on the left, and the region analyzed is shown as the yellow box in the image on the right.

matching effect that can be seen around the portrait and can dominate the visual impression. Using this as a reference, we can see that the image showing the banknote after supercritical CO_2 cleaning removed much of the sebum within the banknote, indicating that the cleaning process is not simply a surface effect.

Similar results were obtained in experiments under the same conditions with £5 banknotes in circulation, which were further treated with sebum and subsequently cleaned with SCCO2. Figure 9 shows both the banknote images and corresponding reflectance data of those banknotes.

On the basis of these results, we find that the supercritical CO_2 cleaning process with ultrasound and no mechanical agitation effectively removes oxidized sebum from United States banknotes. On the basis of spectral changes, the process solubilizes of between 70% and 100% of the deposited sebum layer and appears to preferentially remove moieties responsible for absorption in the 500−650 nm region, which are likely to be the larger fatty acid components of the mixture. In a later section of the paper, we present gravimetric analysis of material removed from banknotes in circulation.

As another demonstration of the effectiveness of supercritical CO_2 cleaning of banknotes, we tested the process on banknotes soiled with motor oil (e.g., Shell ASE 30). The images in Figure 10 clearly show the efficacy of the cleaning process, and the data in Figure 11 show the diffuse reflection spectra before and after cleaning.

Similar results were obtained in experiments with £5 banknotes in circulation, which were further treated with the

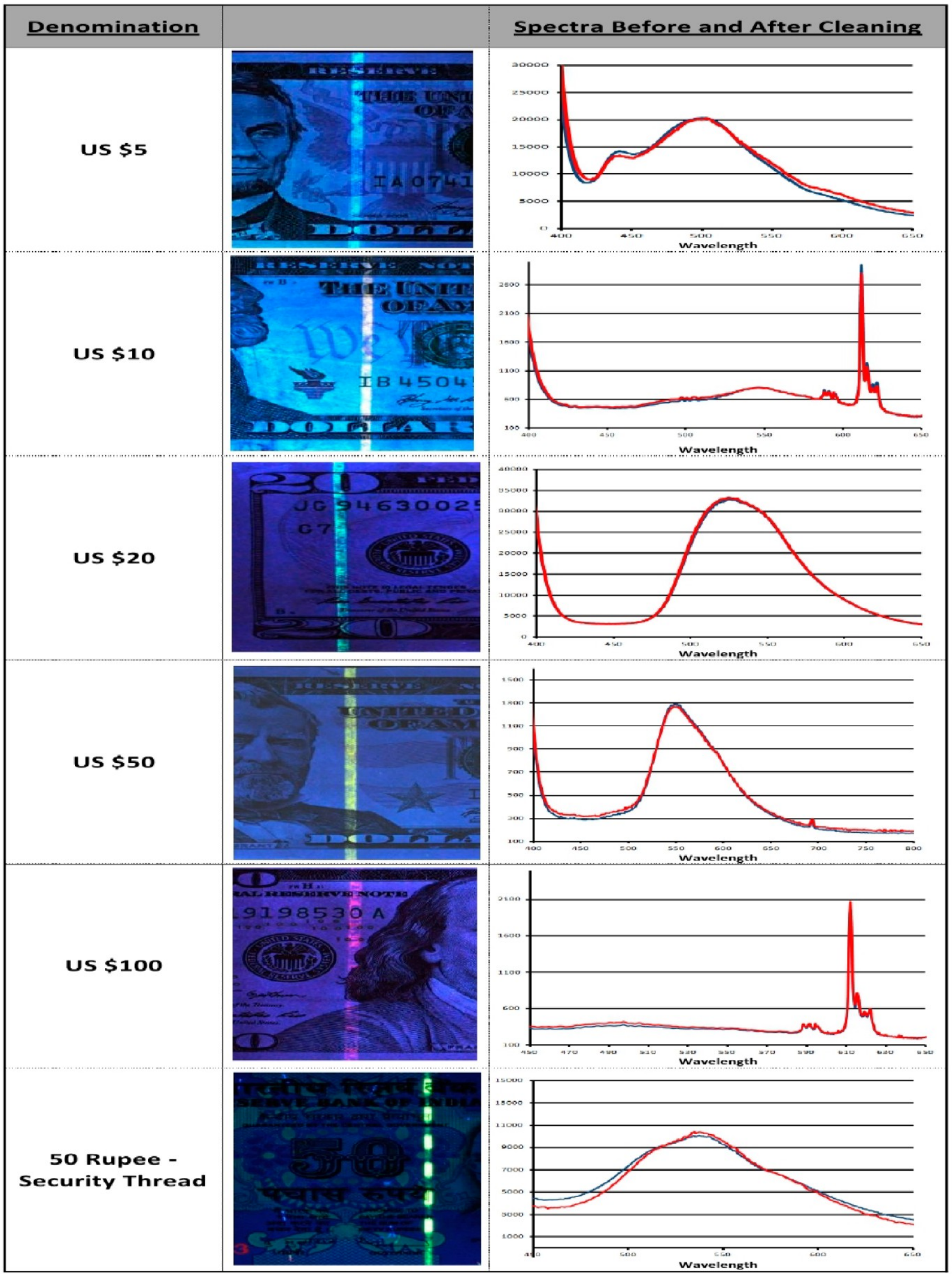


Figure 16. Chart showing the data on UV-excited emission of security threads in United States banknotes and the rupee. The emission spectra before (blue lines) and after (red lines) cleaning are also shown.

motor oil and subsequently cleaned with SCCO2. Figure 12 shows both the banknote images and corresponding reflectance data of those banknotes.

■ BATCH CLEANING OF BANKNOTES IN CIRCULATION

A second generation vessel was used for experiments on cleaning straps of banknotes taken from circulation. These straps, which are created in the central and commercial bank processing environment, contained 100 banknotes with a very high visual level of natural soiling and of the same denomination (either $5 or $1) held by a paper band. The ability to use supercritical fluid cleaning of strapped and bundles notes provides many logistical advantages and eliminates the need for new equipment that would have to be developed to load millions of individual notes into slots for cleaning.

Quantitatively, the level of soiling of the banknotes was classified before and after SCCO2 treatment using a De La Rue Cobra banknote sorting machine equipped with a Soil Detector 5000. This detector and sorting machine measure a banknote's length-average diffuse reflectance on each side of the note at a speed of 2.5 m per second. In the first experiment, one strap of $5

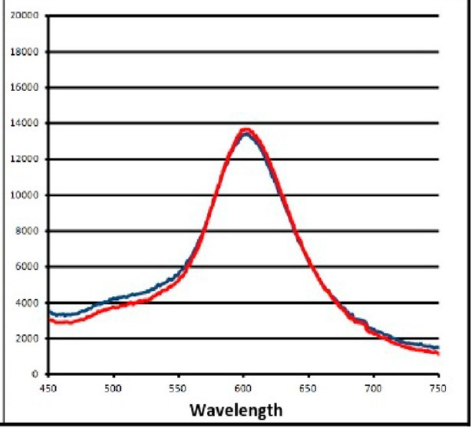
Denomination		**Spectra Before and After Cleaning**
100 Rubles - Security Fibers (Red)		

Figure 17. Security fibers in the Russian ruble with the emission spectra before (blue lines) and after (red lines) cleaning.

banknotes was processed at 60 °C and 5000 psi for 3 h. In the second experiment, one strap of $1 banknotes was processed under the same conditions. During processing, the vial containing the extract was weighed every 15 min. Figure 13 shows the weight of the extract as a function of cleaning time for the $5 strap.

The weight of the $5 strap before cleaning was 99.15 g and after cleaning was 94.26 g, which resulted in 3.78 g of extracted material. We believe the unaccounted 1.11 g was water content in the banknotes that was entrained as vapor with the CO_2 that flowed out of the cell.

The results of the soiling-level classification of the processed banknotes is shown in Figure 14 for the portrait and reverse sides of banknotes for both the $5 and $1 straps. The data shows that strap cleaning is efficient and provides a straightforward approach for cleaning in a bank-processing environment.

■ SECURITY FEATURES

The key to the viability for recycling of soiled banknotes using this technique is the removal of the oxidized oils and other contaminants while leaving a dry banknote and also maintaining the integrity and usefulness of the important and costly public and machine-readable security features.

Optical studies of all of the banknotes tested revealed that no change in the quality or contrast of the printing was observed at all printing size scales after cleaning. This includes the flexographic, gravure, intaglio, and optically variable inks. Figure 15 shows optical contrast measurements that quantitatively confirm this observation.

In addition, none of the polymer-based security features such as holograms and security threads were affected, although in some instances, the windowed security thread in 100 yuan separated from intimate contact with the paper. This occurred sporadically in some notes, while the thread remained intact and an integral part of the note. On the basis of the lack of such an effect in other banknotes tested, this can be remedied by the use of a different adhesive for the thread.

An important public security feature used on nearly all banknotes is long UV (~365 nm) excited fluorescence and phosphorescence. Such fluorescence is easily and economically excited by UV sources that are battery powered and use lamps or LEDs. They are typically used in commercial banks and retail environments as well as in some single note acceptor devices.

Emissive security features are present in many forms in a banknote, including security threads (e.g., United States $5–$100), inks, planchettes, holograms, and fibers. Figure 16 shows that the optical emission spectra of all of the United States security threads as well as those in the Rupee remained intact and maintained their emissive properties throughout 16 h of cleaning with supercritical CO_2 at 60 °C and 2000 psi. This is clearly a limited set given all of the different world banknote designs, but it does show that methods of manufacturing exist that are compatible with the SCCO2 cleaning process.

In addition to emissive security threads, we examined embedded polymeric security fibers such as those typically found in many of the world's banknotes. As an example, we studied the effects of the cleaning process on the fibers in the Russian ruble. The data shown in Figure 17 illustrates the robustness of these UV-excited emissive features against the cleaning process.

Long UV-excited emissive security features are often printed on a banknote using lithographic, flexographic, gravure, and intaglio methods. Examples of this are the yuan, euro, and British pound. We studied printed emissive features in these as well as in other currencies and found most of them to be highly robust as shown by the data for the Chinese yuan in Figure 18.

Similar experiments shown in Figure 19 with United Kingdom banknotes that have a two-color UV emissive pattern revealed that these pigments were partially dissolved away after cleaning. Experiments using only thermal exposure confirmed that this was either dissolution or reaction with the CO_2 and not the thermal degradation of the fluorophore or phosphor.

It is clear from the resilience of the yuan example that inks can be formulated to be resilient to the process of cleaning but that some of the existing ink bases currently used are not.

■ MACHINE-READABLE SECURITY FEATURES

Machine-readable security features play an important role in banknote security. The most common machine-readable security features are based on magnetic susceptibility and capacitance and are most often utilized in single note acceptor applications from ATMs to bill acceptors and vending machines.

The magnetic inks utilized in a number of banknotes, particularly the United States banknotes as well as euro

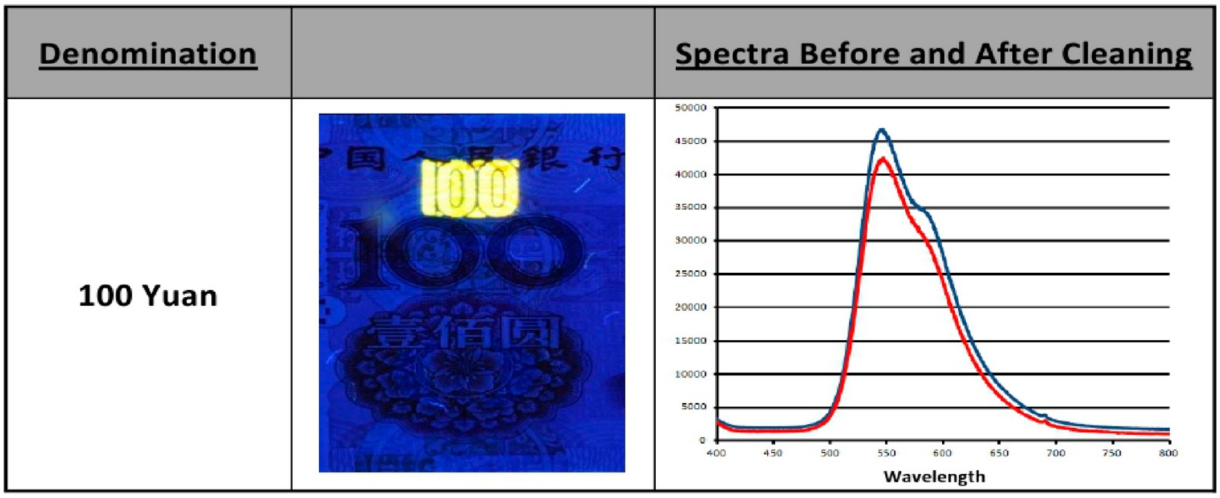


Figure 18. Fluorescent ink features in the Chinese 100 yuan. The emission spectra before (blue lines) and after (red lines) cleaning are also shown.

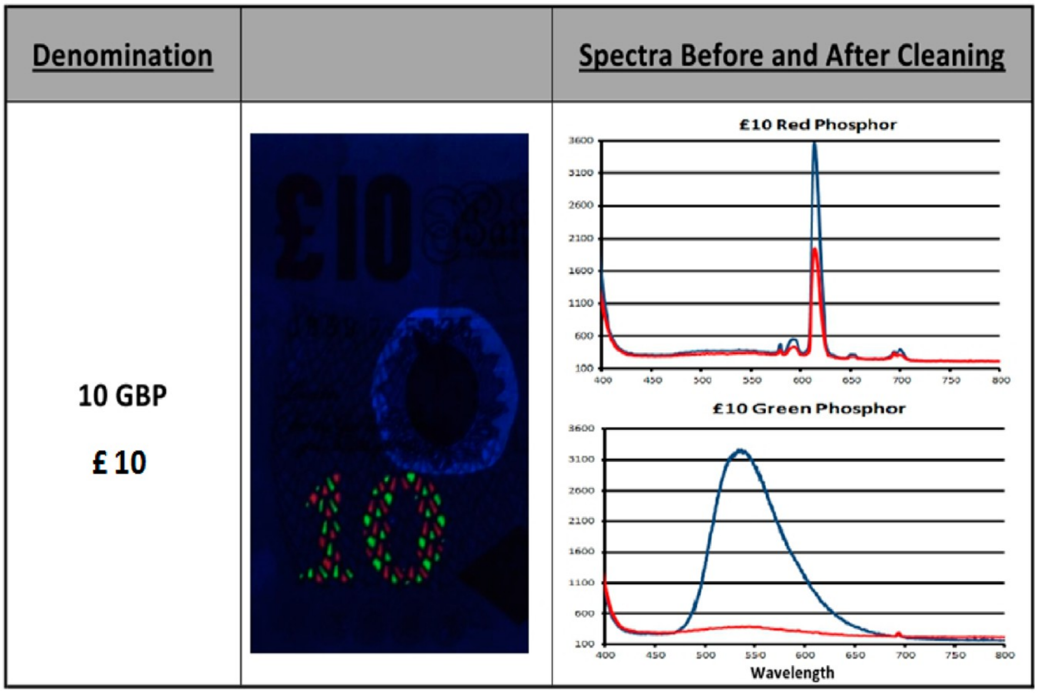


Figure 19. Two color UV-excited ink pattern in a 10 GBP note. The emission spectra before (blue lines) and after (red lines) exposure to supercritical fluid CO_2 cleaning at 60 °C at 2000 psi.

banknotes, were found to be robust and unchanged by the SCCO2 cleaning process at 50 °C and 2000 psi for 16 h. Capacitive machine-readable features such as those used in security threads that rely on metallization also survived the same testing.

In addition to the machine-readable features that are known and used in the public domain and by commercial banks, central banks employ one or more covert features that are typically read at rates of up to 40 banknotes/s on high speed sorters. The signatures of these features are only known to the central banks, enforcement authorities, and companies that supply them. One of these covert security features is Enigma and is supplied by De La Rue plc. Banknotes containing this high-level security were cleaned using the process described and tested using De La Rue sensors. These tests showed that they were completely resilient and unchanged. The widely used M-Feature from Geisecke and Devrient GmbH has not been tested, but given that it is an inorganic oxide, we believe that it will also be resilient and survive the cleaning process.

■ POROSITY

Another important parameter used to determine the fitness of banknotes is limpness. When banknotes have been in circulation, the mechanical wear from folds, handling, and use in bill acceptors results in a loss of mechanical elasticity, which leads to the notes becoming limp. This "limpness" has been shown to be directly related to changes in the porosity of the banknote with mechanical wear. The porosity of the banknotes increases with use and manifests itself in a lower effective elastic constant. Limpness is measured in automated sorting environments using acoustics and ultrasonic reflection.

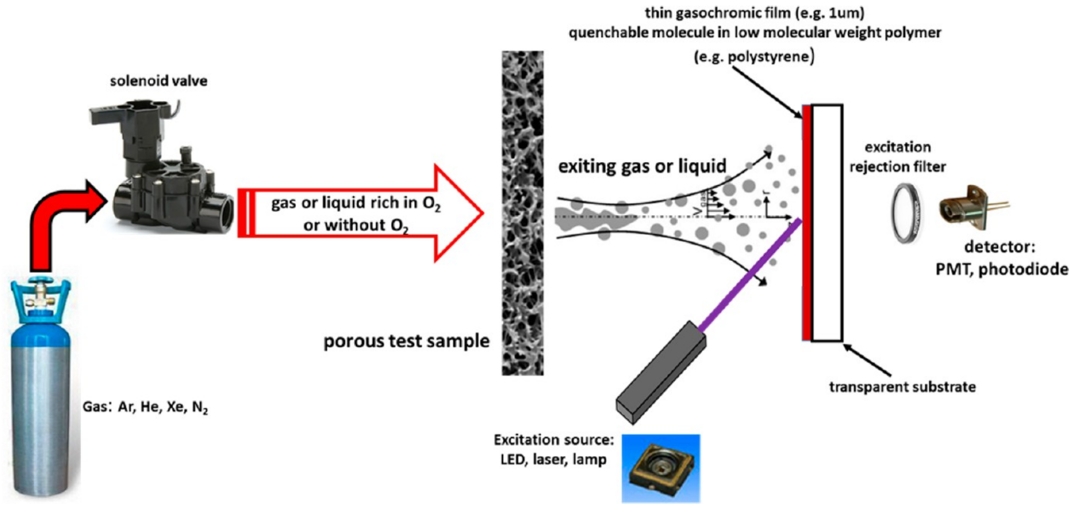


Figure 20. Schematic diagram of the gasochromic-based setup for measuring relative porosity changes.

We measured the porosity of banknotes to determine the effects of SCCO2 cleaning at elevated temperatures on the substrate. It is conceivable that SCCO2 could cause swelling of the fiber network that could exhibit a hysteresis and leave the banknotes more porous. It is also possible that because paper is a nonequilibrium network then the relaxed post-SCCO2 treatment could be compacted relative to the initial state.

The relative porosity was measured using a photoporosimeter developed in-house that allowed for the determination of the changes in the cellulosic network caused by the SCCO2 cleaning process on United States banknotes. The porosimeter was based on a response of a gasochromic layer of material (Pt-porphyrin in low density polystyrene) to a solenoid-triggered pulse of argon gas. The pulse of argon gas (250 ms) flows through the banknote and displaces ambient oxygen creating a transient increase in the UV fluorescence (680 nm) of the UV-illuminated gasochromic film. In effect, the delay time observed is a measure of the void fraction and tortuosity of the porous banknote. Figure 20 shows a schematic of the porosimeter setup that has a measured response time of 10 ms.

Figure 21 shows the gas transmission characteristics of a worn \$1 note in circulation that exhibits a delay relative to the trailing edge of the gas pulse released by the solenoid shown by the yellow trace. The delay detected at the gasoshromic layer is ~50 ms, which is five times longer than the system response time.

Figure 22 shows the signals of the uncirculated note before and after SCCO2 cleaning. The results indicate that the uncirculated note has lower porosity, resulting in both a diminished signal and a longer delay (~250 ms) relative to the trailing edge of the argon gas pulse. The figure also shows that the signal of the uncirculated note after SCCO2 cleaning resulted in a ~200 ms delay and ~30% increase in gas transmission. This small change is not significant when compared to a ~50 ms delay and ~20-fold increase in gas transmission of a worn note in circulation and indicates that the process has no significant effect on the porosity of the notes.

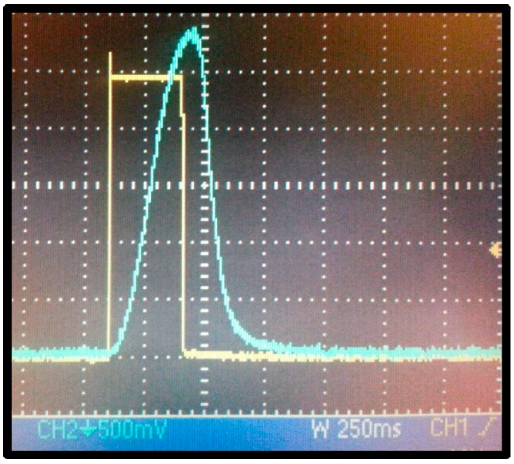


Figure 21. Transient response to argon gas penetrating a United States \$1 note in circulation. The yellow trace shows the solenoid opening and closing trigger pulse, and the blue trace shows the fluorescence response of the gasochromic film. Time scale is 250 ms/div, and the signal levels are in 500 mV/div.

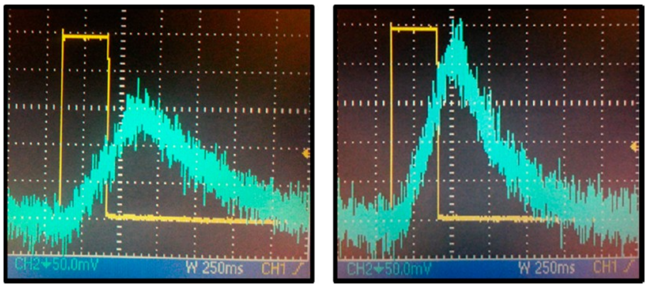


Figure 22. Transient response to argon gas penetrating a United States \$1 uncirculated note before (left) and after SCCO2 cleaning (right). Time scale is 250 ms/div, and signal level is 50 mV/div.

■ REMOVAL OF ORGANISM COLONIES

In addition to the removal of sebum and other soiling agents, we also tested the banknotes to determine the efficacy of the process in removing common organism colonies from banknotes. Swab tests were conducted on United States \$1 bills and grown in TSAB/Mac Conkey media at 35 °C. The tests revealed that colonies of both *micrococcus luteus* (1 colony/swab) and yeast (234 colonies/swab) were present with the prevalent organism being the latter. Subsequent to SCCO2 cleaning, the colony counts were zero for both types of organisms. Although cleaning of banknotes does not keep them clean once they are returned into circulation, it does allow for the mitigation of the spread of disease to individuals processing currency.

CONCLUSIONS AND FUTURE WORK

We have shown that supercritical CO_2 is an effective cleaning process for the removal of oxidized sebum, the primary soiling component of banknotes. In addition, the process was effective in removing other soiling agents such as motor oil and common organism colonies. The process was shown to be effective, while at the same time not damaging the printed features at all spatial levels or the expensive security features in banknotes. Examples of all security features, including threads, lenticular arrays, watermarks, UV emissive inks, and fibers, all survived and remained effective after cleaning. Machine-readable features such as magnetic inks and capacitive structures also were left intact. In addition, the Enigma level III feature was also tested and exhibited no change in performance, and we expect similar results for the M-Feature.

The work also demonstrated that banknotes in circulation could be cleaned while in conventional 100 note straps, typical of the commercial and central bank environment. Data on the distribution of banknote soiling levels before and after cleaning in straps showed a significant decrease in soiling levels on both sides of the notes. These results confirm that supercritical fluid cleaning is practical and can have dramatic consequences for central bank budgets as well as mitigating the environmental impact of the disposal of unfit banknotes.

Future work will focus on conducting large volume (>100,000) testing of notes in circulation using a 200 L closed loop system (CO_2 recycling) combined with central bank processing of notes before and after cleaning in order to more accurately assess the impact of this approach to reducing the cost of replacement banknote production and environmental disposal at the processing nodes of the banknote cycle.

AUTHOR INFORMATION

Corresponding Author

*E-mail: nlawandy@spsy.com.

Notes

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