*J. Cosmet. Sci.*, 51, 275–287 (September/October 2000)

# Advancements in spectroscopic and microscopic techniques for investigating the adsorption of conditioning polymers onto human hair

JAMES S. DALTON, GEOFFREY C. ALLEN, PETER J. HEARD, KEITH R. HALLAM, NICK J. ELTON, MATTHEW J. WALKER, and GARY MATZ, *Interface Analysis Centre, University of Bristol, 121 St. Michaels Hill, Bristol BS2 8BS, UK (J.S.D., G.C.A., P.J.H., K.R.H.), Imerys Central Research, Par Moor Laboratories, St. Austell, Cornwall PL24 2SQ, UK (N.J.E, M.J.W.), and Calgon Corporation, P.O. Box 1346, Pittsburgh, PA 15230 (G.M.).*

*Accepted for publication August 11, 2000.*

Synopsis

Adsorption of the polyquaternium-6 (Merquat®-100) conditioning polymer onto bleached human hair has been investigated using environmental scanning electron microscopy (ESEM), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SIMS). ESEM is not limited to a high-vacuum environment, and therefore hair morphology could be studied under ambient and hydrated conditions. XPS gave elemental analysis on the surface of the hair, in addition to information on the chemical environment of the surface atoms. SIMS can produce high-resolution ion distribution images on the hair's surface. Both XPS and SIMS detected carbon, nitrogen, oxygen, and sulfur on the surface of the untreated bleached hair, all of which were attributed to the biological matrix. Silicon was also detected and its presence was attributed to either a previous silicone cosmetic application or to surface contamination. Due to the similar elemental composition of hair and Merquat-100, treatment of the hair with a phosphorus-labeled polyquaternium-6 cationic conditioning polymer was also investigated. XPS, with sensitivity of 1000 parts per million (ppm), could not detect phosphorus present from any adsorbed polymer, but SIMS, with 1–10 ppm sensitivity, allowed high-resolution images to be obtained that illustrated the adsorption of polymer onto the hair's surface.

## INTRODUCTION

Due to the increasing complexity of personal care formulations and strong market forces, there is now a greater necessity than ever for manufacturers to substantiate claims concerning their products. Therefore, there is a current need for further development of analytical techniques capable of giving biological and chemical information on both the hair's surface and adsorbed cosmetic molecules such as polymers. The aim of this paper is to demonstrate how techniques such as X-ray photoelectron spectroscopy (XPS), secondary ion mass spectrometry (SIMS), and environmental scanning electron micros-



Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

copy (ESEM) can be exploited to this end. These techniques have the advantage over many indirect and macroscopic tests that give little information on hair morphology and the presence of specific adsorbates such as polymers. Quaternized cationic polymers have a high affinity for the hair's surface due to the hair's negative charge at normal pH and are typically used as conditioners due to their high cationic charge density. Generally, hair conditioning involves some interfacial alteration in the protein matrix of the hair.

There is a wealth of literature covering electron microscopy studies of hair (1). Studies include ESEM, which, unlike traditional SEM, does not require a high vacuum and can allow the hair to be studied in an ambient and hydrated environment (2). More recently, attention has been paid to hair structure and morphology using scanning probe and atomic force microscopy (SPM and AFM), allowing the hair to be imaged at varying pH and hydration levels (3–6). SPM has also been instrumental in detecting cationic polymer adsorption onto hair, these polymers adsorbing strongly at normal pH due to high cationic charge density, an essential property for hair conditioning (7,8).

There is little literature on spectroscopic studies of hair; however, electron energy loss spectroscopy (EELS) has been used to map the sulfur distribution in human hair, giving information on the distribution and abundance of keratin bonds (9). In addition, SIMS imaging has recently been proposed by Gillen *et al.* as a promising technique for mapping distributions of certain ions in hair such as $Ba^+$, $K^+$, and $C_2H_3^+$ after doping hair in various inorganic and organic solutions. Their recent work gives some good examples (10).

With the emphasis on cosmetics, this paper shows how a variety of techniques can be used to investigate the presence of adsorbed species onto biological materials such as hair.

## MATERIALS AND METHODS

### GENERAL HAIR TREATMENT

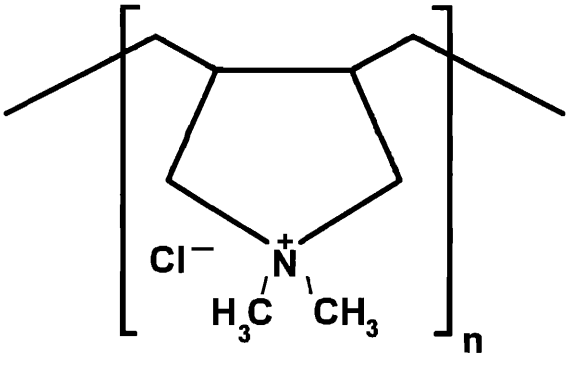
Bleached hair samples were purchased from DeMeo Brothers, New York. The bleached hair was studied both untreated, by simply rinsing the sample in distilled water and allowing to dry naturally, and treated, as outlined below.

Treatment was carried out using polyquaternium-6, a cationic conditioning polymer, [poly(dimethyldiallyl ammonium chloride), Merquat®-100, 40% active, Calgon Corporation, for polymer composition (see Figure 1a)] by soaking in a 0.1% w/w aqueous solution (18MΩ deionized water) for two hours and then rinsing in running deionized water for two minutes. The tress was then left to dry naturally for 24 hours.

To enable clearer detection of the polymer adsorption, and to differentiate the polymer from the hair with greater distinction, some tresses were treated with a phosphorus-labeled polyquat-6 derivative. This phosphorus-labeled Merquat® 100 was prepared by polymerizing dimethyldiallylammonium chloride in the presence of hypophosphorous acid as a chain regulator. The structure is given in Figure 1b. For distinction, the commercially available Merquat®-100 will be referred to as N-Merquat®-100, and the synthesized phosphorus-labeled as P-Merquat-100.

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

(a)




(b)

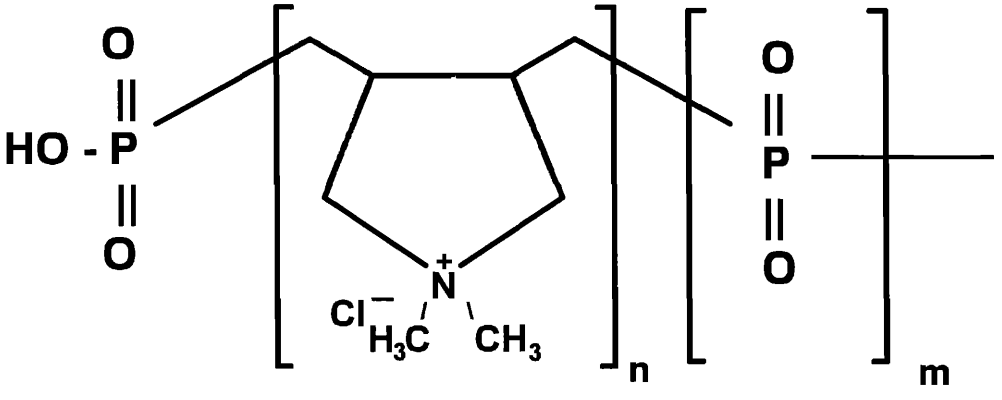



**Figure 1.** a: Structure of the polyquaternium-6 polymer. b: Structure of the phosphorus-labeled polyquaternium-6 polymer. The molar ratio of n:m is approximately 99:1. For polymer synthesis see text.

INSTRUMENTATION

*Environmental SEM.* Environmental SEM (ESEM) permits high-resolution imaging of fully hydrated materials. ESEM offers all the capabilities of conventional SEM, but a differential pumping system enables specimens to be maintained in a gaseous environment to around 20-torr pressure (11). Secondary electron imaging exploits the ionized gas in the chamber by using proportional gas amplification of the SE signal (12). The ionized chamber gas also dissipates charge buildup on the specimen. Water vapor is a convenient and efficient imaging gas. By balancing chamber pressure and specimen temperature along the 100% RH isobar, specimens may be maintained in a fully

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

hydrated state for long periods of time. Resolution is comparable to a conventional SEM (~5 nm with a tungsten gun). Techniques like energy dispersive spectroscopy (EDS) are routinely employed, but there are special considerations due to the gas in the chamber (13,14). There is little literature on ESEM applications to human hair, but related studies include the study of wool (15) and the development of detergents and personal products (16).

In this work a Philips-Electroscan ESEM 2020 was used with a PGT IMIX EDS system. Hair was clamped in a special metal stub designed to hold hair strands flat and to allow reproducible positioning in the ESEM. In this way, individual strands could be examined before and after treatment with conditioning polymers. The stub was temperature-controlled via a Peltier stage. Specimens were typically imaged at ~5°C and chamber pressure ~5 Torr to maintain approximately 100% RH.

*X-ray photoelectron spectroscopy (XPS).* XPS analyzes the near-surface of materials with analysis depths up to 50 Å. It can give quantitative chemical information as well as oxidation and structural environments on all elements apart from hydrogen and helium. Soft X-rays, of energy $h\nu$, excite valence electrons from valence and core orbitals of surface atoms. This is shown in Figure 2. The kinetic energy of the ejected electron, $E_K$, is measured by an electron energy analyzer. These photoelectrons have energies according to the relationship (17)

$$E_B = h\nu - E_K - \Phi$$

where $\Phi$ is the work function of the spectrometer. $E_B$ is the binding energy of the photoelectron to the parent atom, and this can be calculated from the other measured and known values. XPS spectra are traditionally plotted as $E_B$ vs photoelectron intensity. XPS peaks can then be identified using tabulated binding energy values from XPS handbooks yielding information on chemical composition and bonding environments.

Tresses of untreated and polymer-treated hair were clamped into separate metal stages such that a mesh of hair at least 5 mm by 5 mm could be studied. XPS spectra were acquired using a Fisons ESCAscope photoelectron spectrometer with dual anode X-ray

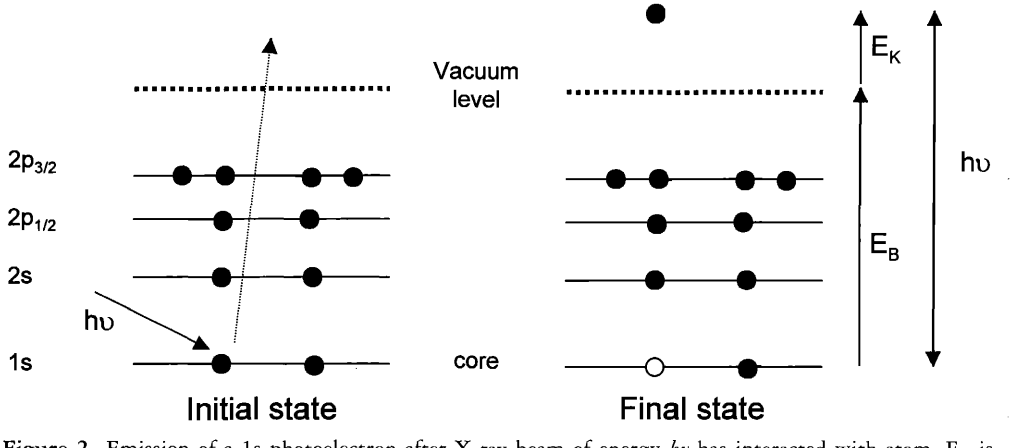



**Figure 2.** Emission of a 1s photoelectron after X-ray beam of energy $h\nu$ has interacted with atom. $E_B$ is electron binding energy and $E_K$ the kinetic energy after leaving vacuum level.

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

sources (Mg/Al) and a concentric hemispherical electron energy analyzer. Data acquisition and manipulation was carried out using microprocessor units with VGS software. During acquisition of spectra the pressure in the main chamber was maintained at $8 \times 10^{-9}$ torr to ensure a clean sample surface. Charge referencing was carried out against adventitous hydrocarbon (C1s 284.6 eV) (17) from pump oil contamination.

*Secondary ion mass spectrometry (SIMS).* SIMS allows a mass spectrum of elemental and molecular species present in the surface to be obtained (18). A highly focused ion beam penetrates the surface under study, sputtering atoms and ions in the uppermost surface layer, which are then analyzed according to their mass/charge (m/z) ratio by a mass spectrometer. In a manner similar to SEM imaging, high-resolution SIMS images were obtained by raster scanning the beam over the desired area on the hair's surface and detecting locations of sputtered ions having a prefixed m/z value. However, in contrast to XPS, SIMS is a surface-destructive technique due to the sputtering or erosion of the surface atoms, and therefore acquisition times for spectra and images were kept to a minimum.

The SIMS spectra and images were obtained using an in-house instrument comprised of an electronically variable aperture-type gallium ion gun (FEI SD Gallium LMIS EVA focusing column) fitted to a double-focusing magnetic-sector mass analyzer (Vacuum Generators model 7035). An Everhart-Thornley electron detector was used for acquisition of secondary electron images. For secondary ion analysis, a 1nA, 25keV impact $Ga^+$ microbeam was used, which allowed an image resolution of 300 nm. The upper mass limit was configured at 100 daltons (D), and the instrument was calibrated using values of 68.926 and 70.925 D for the backscattered $Ga^+$ ions. Analysis was carried out using the "Dayta" software package (19) running under Windows 95.

For obtaining spectra of the hair's outer surface (epicuticle), 10-mm hair lengths were mounted over an aluminium stub using silver dag adhesive at either ends of the hair. It was essential that the hair made good contact with the stub throughout its length, and so an aluminium mesh was placed over the sample to essentially clamp the hair and enable it to lie flat. Due to problems of imaging hair lengths, which will be outlined later, SIMS images were obtained on hair cross sections. Here 10-mm-length samples of both N- and P-Merquat-100–treated hair were embedded in separate blocks of Agar 100 epoxy resin and then microtomed with a diamond knife. Due to the insulating nature of the resin and hair, the blocks were gold-coated for analysis to eliminate charging. Careful coating of the hair under high vacuum would not affect the elemental distribution, and the metal layer was etched away before analysis using the 1nA 25keV $Ga^+$ microbeam.

## RESULTS AND DISCUSSION

### ESEM

Figure 3a shows an ESEM image of virgin bleached hair. Figure 3b shows hair from the same batch after exposure to pH 9.5 at 20.9°C. Some roughening and raising of the cuticle is visible. Figure 3c shows hair from the same batch after treatment with a 3% aqueous solution of Merquat-100 at pH 7. Although a little raising of the cuticle still seems to be present after treatment with Merquat-100, this may be expected, as the hair

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

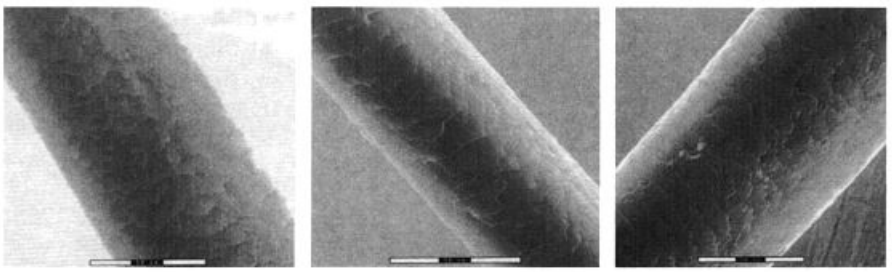



**Figure 3.** a: Untreated virgin bleached hair. b: Virgin bleached hair exposed to aqueous environment at pH 9.5 and 29.9°C. Some raising of the cuticle is visible. c: Hair treated with 3% Merquat-100 at pH 7. Conditioning treatment is expected to smooth and lower the cuticle in damaged hair.

was treated only once with the polymer solution to allow fair comparison to the other techniques. However, the many images obtained from both untreated and treated hair showed less roughening on the conditioned cuticle compared to the untreated. While ESEM is useful for revealing surface topography of the hair, it was not useful for determining the distribution of Merquat over the surface owing to lack of topographic or Z contrast (except at unreasonably high polymer doses). Hair treated with N-Merquat and P-Merquat was examined using EDS. The elemental composition of N-Merquat was not sufficiently different from that of the hair to allow EDS mapping of the polymer distribution. In principle, the theoretical level of P in the P-Merquat ought to be high enough to allow detection by EDS. However, in practice, the concentration of P over the surface of P-Merquat–treated hair was below the detection limit, and this approach was abandoned in favour of SIMS and XPS. Beam damage can be a problem in ESEM, especially with hydrated biological materials during EDS (14). In this study, beam damage to the hair did not appear to occur under normal operating conditions.

### XPS

The XPS spectrum of untreated hair is given in Figure 4. This shows the presence of carbon, nitrogen, oxygen, sulphur, and silicon from the outer surface of the epicuticle. The first four elements were expected, as they form the biological matrix of the hair, the epicuticle being rich in the amino acid cysteine, which contains keratinized disulfide bonds that give the hair its strength (1). Silicon on the surface of hair can be explained by the presence of silicon polymers in many shampoo and conditioner formulations (20), and the hair may have been treated with these prior to study. In addition, it is commonly known that silicones contaminate the surface of most materials, in particular polymers (21), which makes any surface study of silicon difficult. Peak fitting was carried out on regional elemental spectra to obtain information on the chemical environment. The carbon 1s photoelectron spectra was resolved to two peaks, with the most intense due to both the hydrocarbon chains present in the protein and pump oil contamination. The less intense peak was due to the carbon from the carboxylic groups in the protein matrix. The nitrogen and oxygen 1s peaks at 399.9 eV and 531.8 eV, respectively, were characteristic of the organic biological matrix formed from the amino acids (22). For the

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

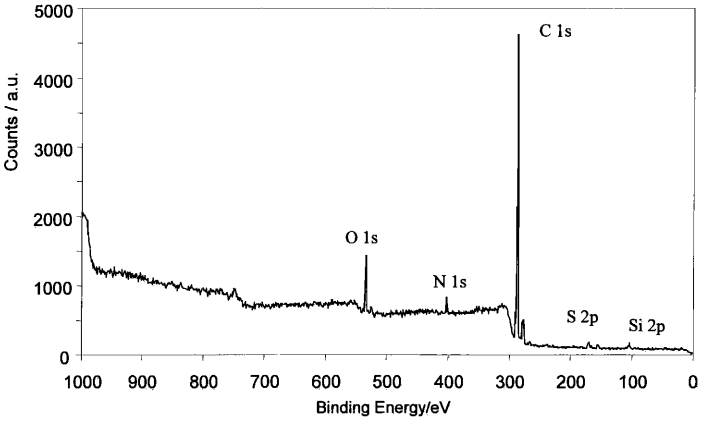



**Figure 4.** Wide-scan XPS spectra of bleached hair.

silicon 2p peak, regional spectra showed a singlet at 101.9 eV, confirming the presence of silicones or silanes on the surface of the hair (22).

Analysis of the spectra from the N-Merquat®-100 and P-Merquat®-100–treated hair showed no new peaks compared to the untreated. It appeared that XPS was not sensitive enough to detect any additional phosphorus or chlorine that would be present due to polymer adsorption. A more sensitive technique is therefore needed to detect any adsorbed polymer, and whereas the sensitivity for XPS is around 1000 ppm, SIMS enables the detection of most elements down to the 1–10 ppm scale.

### SECONDARY ION MASS SPECTROMETRY (SIMS)

*SIMS spectra of hair lengths: Untreated hair.* A negative SIMS spectrum of an *untreated* hair's outer surface is given in Figure 5a. The instrument was configured so that only ions sputtered from the hair were detected. The acquisition time for spectra was approximately two minutes, and this sputtered less than 10% of the thickness of the epicuticle. In agreement with the XPS measurements, $C^-$ (12D), $CH^-$ (13D), $O^-$ (16D) $OH^-$ (17D), $C_2^-$ (24D), $C_2H^-$ (25D), $CN^-$ (26D) and $S^-$ (32D) peaks were observed, these making up the biological matrix at the surface. Of particular interest in the spectrum are the peaks at 19 D ($F^-$) and 35/37 D ($Cl^-$), which were undetected using XPS. These are therefore present in small amounts at the surface (<1000 ppm); however, it should be noted that the relative intensities of the peaks for the different species in SIMS cannot be compared directly, as the sensitivity factors of the elements vary greatly. It is thought that the chlorine may originate from the bleaching process. An explanation for the presence of fluorine is that the hair may have been treated prior to study with a cosmetic containing a fluorocarbon surfactant or polymer, which are used due to their very low surface free energies. A positive ion spectrum was also acquired, although, apart from sodium (23D), potassium (39D) and silicon (28D) peaks, there was nothing further of interest. Traces of sodium and potassium were detected due to their very high sensitivity in SIMS and are present as trace metals in the hair.

*SIMS spectra of hair lengths: P- and N-Merquat®-100–treated hair.* The negative SIMS spectrum of hair treated with N-Merquat®-100 is given in Figure 5b. This spectrum is

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

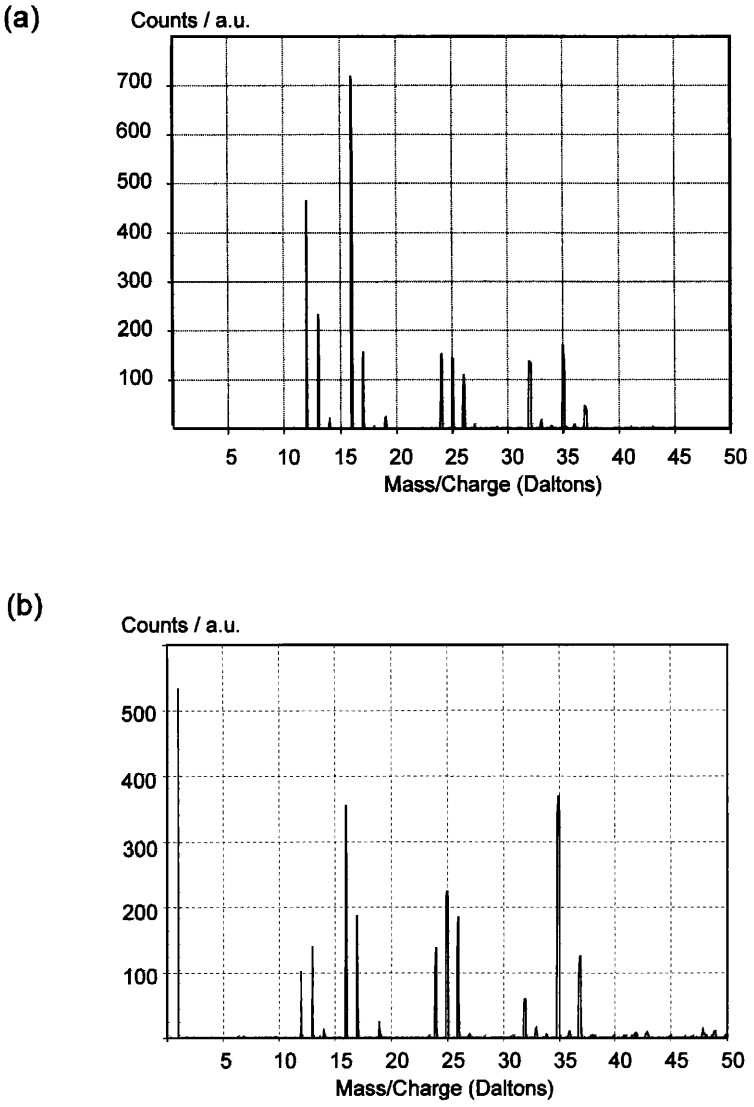



**Figure 5.** a: Negative ion spectrum of untreated bleached hair. Counts are in arbitrary units. b: Negative ion spectrum of N-Merquat®-100–treated hair. Counts are in arbitrary units.

similar to that of the *untreated,* although the relative amount of chlorine on the hair appears to be higher. The presence of chlorine in the polymer may be responsible for the observed higher count rate, although from this evidence alone it is not yet possible to draw firm conclusions.

Elemental and molecular SIMS imaging was initially carried out on the hair lengths, although due to the hair not lying flat on the stub, image quality was poor and much of the contrast in these images is related to the topography of the specimen. Therefore, imaging was carried out on hair cross sections by embedding in resin as detailed previously. Figure 6a shows the microtomed surface of the resin and hairs obtained by

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

light microscopy. Figure 6b shows a typical hair that was studied, clearly exhibiting the epicuticle, cortex, and medulla. The cross section is slightly oval in shape, as the hair has been embedded at a slight angle to the normal cut.

### ELEMENTAL AND MOLECULAR MAPPING OF HAIR CROSS SECTIONS

Secondary electron and ion images were taken over the cross section of many hairs treated with either N- or P-Merquat®-100, and typical maps are shown in Figures 7 and 8. It was assumed that the elemental distribution remained constant along the hair during imaging. This was supported by etching a further 20 nm into the hair and re-imaging. It was observed that elemental maps were similar to those acquired on the initial surface.

*N-Merquat®-100 hair.* Figure 7a gives the backscattered electron image of a hair embedded in resin. It should be noted that the hair has remained similar in appearance to that taken by the optical microscope, and so it would appear that the effect of ultra-high vacuum on the hair has not affected it too greatly. Figure 7b shows how $CN^-$ (26D) mapping can be used to emphasize the biological matrix, and Figure 7c shows how $O^-$ sites are primarily located in the outer surface of the hair. Figure 7d indicates that the

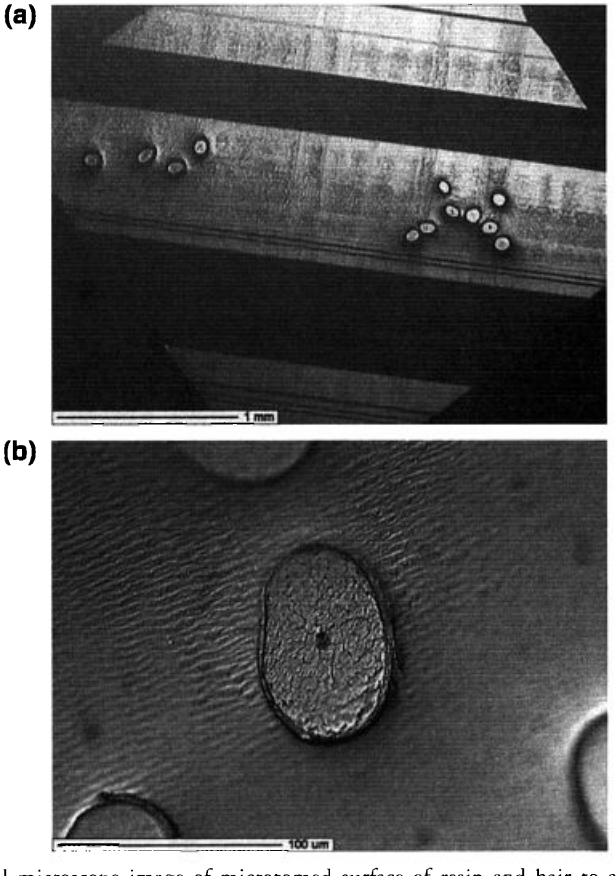



**Figure 6.** a: Optical microscope image of microtomed surface of resin and hair to allow flat hair cross-section studies. b: Optical microscope image of a typical hair cross section under study.

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

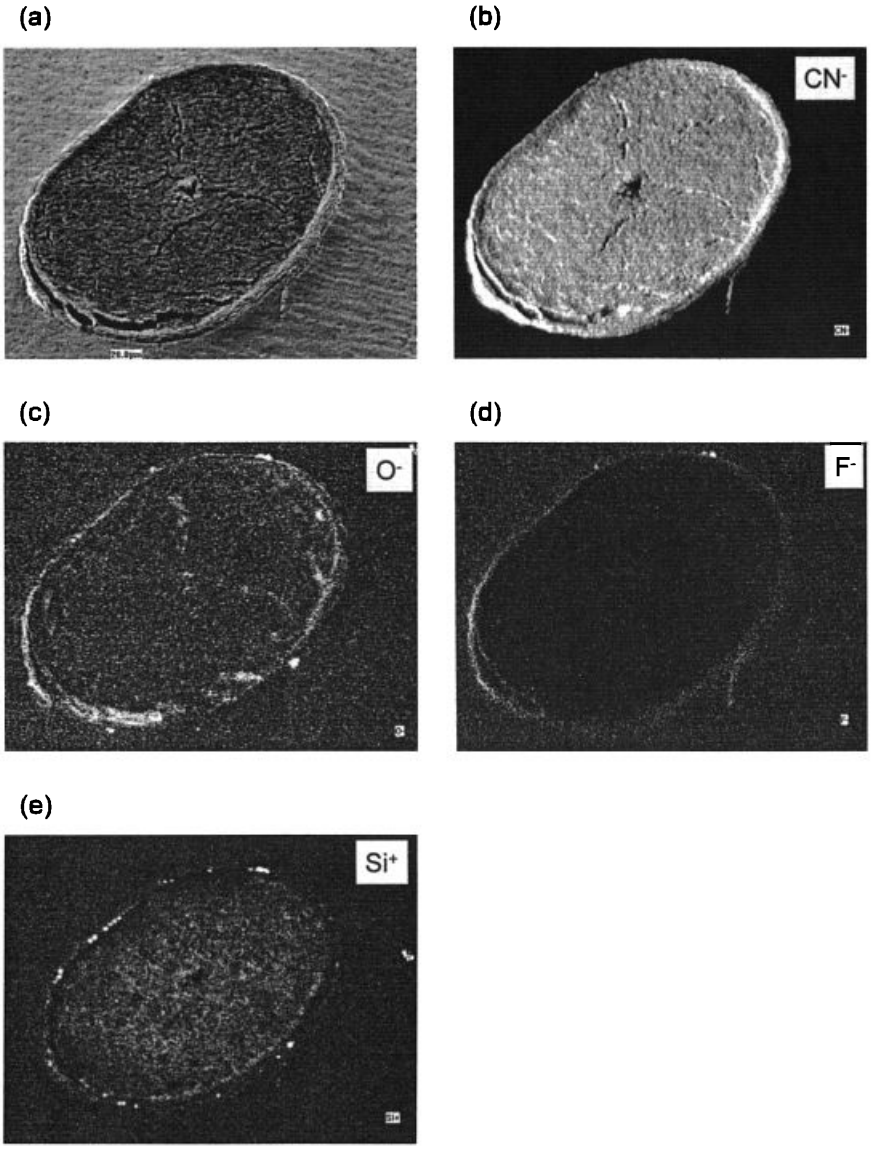



**Figure 7.** a: Backscattered secondary electron image of an N-Merquat®-100–treated hair in cross section embedded in resin. Epicuticle, cortex, and medulla are clearly seen for this hair. b: SIMS image of the $CN^-$ (m/z 26D) distribution in the cross section shown in (a). c: SIMS image of the $O^-$ (m/z 16D) distribution in the cross section shown in (a). d: SIMS image of the $F^-$ (m/z 19D) distribution in the cross section shown in (a). e: SIMS image of the $Si^+$ (m/z 28D) distribution in the cross section shown in (a).

$F^-$ is also predominantly located on the outer surface, which gives evidence that its presence is due to environmental exposure or a cosmetic product. Unfortunately, $Cl^-$ imaging was not possible due to the composition of the resin.

Figure 7e illustrates positive ion imaging, which can be used to detect the presence of metals. The distribution of silicon in Fig 7e is again probably due to a previous silicon-based cosmetic product or surface environmental contamination. Ideally, both

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

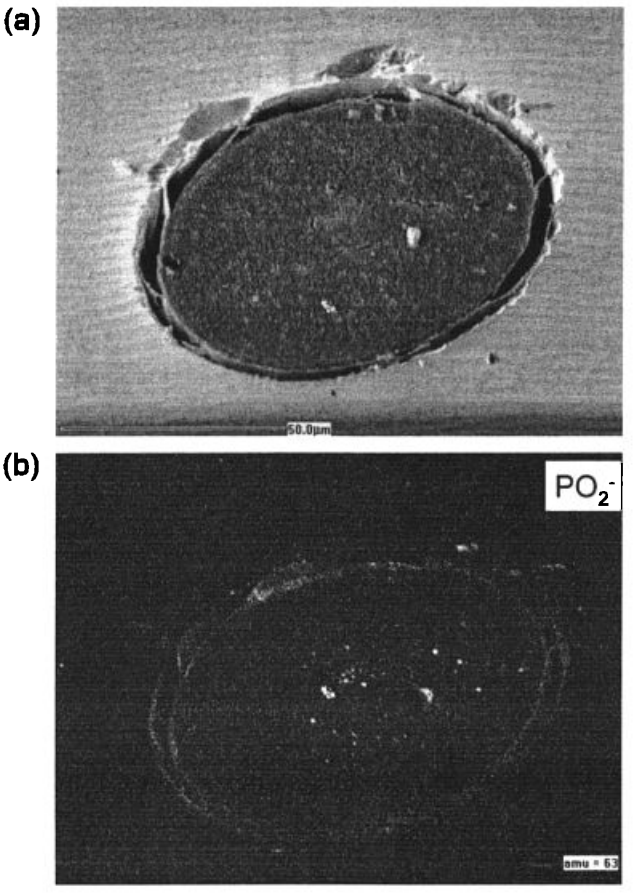



**Figure 8.** a: Backscattered secondary electron image of a P-Merquat®-100–treated hair in cross section embedded in resin. In this hair sample no medulla is present. b: SIMS image of the $PO_2^-$ (m/z 63D) distribution in the cross section in (a).

polymer solution and hair should be washed in hexane for siloxane impurity removal before study, although, of course, this is highly impractical for any cosmetic study. Ideally, silicon mapping should be carried out using isotopic labeling.

It should be noted that as both the biological matrix of the hair and the polymer have similar elemental compositions it was not possible to obtain any distinction between them. It was for this reason that a phosphorus-labeled polymer was synthesized.

*P-Merquat®-100 hair.* The phosphorus labeling of Merquat®-100 allows for unique detection of adsorbed polymer. Figure 8a shows the secondary electron image of a hair. Phosphorus imaging via $PO_2^-$ (m/z 63D) is shown in Figure 8b and shows a weak ion count around the epicuticle, indicative of adsorbed polymer. This was not previously detected with the N-Merquat®-100–coated hair. To ensure that it was $PO_2^-$ imaged at m/z 63D and not other ion fragments from the hair, a process of ion elimination was carried out. Standard spectra of hydrocarbon, chlorinated, polyethylene glycol, and nylon-6 (synthetic, containing peptide links) polymers contained no negative ion fragments at m/z 63D (23). A possible detectable hydrocarbon fragment at m/z 63D is

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

$C_5H_3^-$, but due to the high bond order, this is unlikely. Indeed, an image taken at m/z 65D to map for the similar $C_5H_5^-$ ion showed virtually zero ion count over the resin and hair. It can therefore be concluded that SIMS has the ability to detect adsorbed macromolecules on the surface of hair.

## CONCLUSIONS

Results have shown how these three techniques can be combined to give significant information on the adsorption of polymer onto hair; indeed this can be extended to the adsorption of most other adsorbates onto biological materials such as hair, nails, tissue, teeth, or skin. SIMS allowed direct surface analysis and was capable of detecting adsorbed polymers on the hair's surface. Although direct detection could only be obtained using SIMS, XPS has the ability to define the chemical environment or oxidation state of the elements at the surface of the hair. These surface elements may be part of the hair's biological matrix, surface contaminants, or adsorbate molecules at a particularly high concentration.

The recent advancement of ESEM has allowed biological materials such as hair to be studied without the prerequisite of high vacuum, and therefore the behavior of hair can be observed under ambient and hydrated environments.

## ACKNOWLEDGMENTS

The authors would like to thank Jeremy Hooper, Dave Greenhill, and Martin Taylor of Imerys for useful discussions and assistance with experimental work and sample preparation.

## REFERENCES

(1) C. R. Robbins, *Chemical and Physical Behaviour of Human Hair,* 3rd ed. (Springer-Verlag, New York, Berlin, Heidelberg, 1994).
(2) R. E. Cameron, Environmental scanning electron microscopy in polymer science, *TRIP,* **2,** 116–120 (1994).
(3) J. R. Smith, Use of atomic force microscopy for high resolution non-invasive structural studies of human hair, *J. Soc. Cosmet. Chem.,* **48,** 199–208 (1977).
(4) J. R. Smith, A quantitative method for analysing AFM images of the outer surfaces of human hair, *J. Microscopy,* **191,** 223–228 (1998).
(5) P. Hössel, D. I. R. Sander, and W. Schrepp, Scanning force microscopy: Researchers describe how they use this technology to examine hair treated with cationic polymers, *Cosmet. Toiletr.,* 111, 57–65 (1996).
(6) S. D. O'Connor, K. L. Komisarek, and J. D. Baldeschwieler, Atomic force microscopy of human hair cuticles: A microscopic study of environmental effects on hair morphology, *J. Invest. Dermatol.* **105,** 96–99 (1995).
(7) R. L. Schmitt and E. D. Goddard, Investigation into the adsorption of cationic polymers, *Cosmet. Toiletr.,* **109,** 83–93 (1994).
(8) E. D. Goddard and R. L. Schmitt, Atomic force microscopy investigation into the adsorption of cationic polymers, *Cosmet. Toiletr.,* **109,** 55–61 (1994).
(9) P. Hallegot and P. Corcuff, High spatial resolution maps of sulphur from human hair sections: An EELS study, *J. Microscopy,* **172,** 131–136 (1993).
(10) G. Gillen, S. Roberson, C. Ng, and M. Stranick, Elemental and molecular imaging of human hair using secondary ion mass spectrometry, *Scanning,* **21,** 173–181 (1999).

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)

(11) R. E. Cameron, Environmental SEM: Principles and applications, *Microsc. Anal.,* 11–13 (May 1994).
(12) G. D. Danilatos, Introduction to the ESEM instrument, *Microsc. Res. Tech.,* **25,** 354–361 (1993).
(13) R. B. Bolon, "X-Ray Microanalysis in the ESEM," in *Microbeam Analysis,* D. G. Howitt, Ed. (San Francisco Press, San Francisco, 1991).
(14) D. C. Sigee, Environmental SEM and X-ray microanalysis of biological materials, *Mikrochim. Acta,* 15(suppl), 283–293 (1998).
(15) G. D. Danilatos and J. H. Brooks, Environmental SEM in wool research—Present state of the art, *Proceedings of the 7th International Wool Textile Research Conference, Tokyo, 1985,* **1,** 263–272 (1985).
(16) K. Hoyberg and K. G. Kruza, Application of environmental scanning electron microscopy in the development of detergents and personal products, *Microsc. Res. Tech.* **25,** 424–428 (1993).
(17) D. Briggs and M. P. Seah, Eds., *Practical Surface Analysis, 2nd ed.,* Vol. 1 (John Wiley & Sons, Chichester, 1992).
(18) D. Briggs and M. P. Seah, Eds., *Practical Surface Analysis, 2nd ed.,* Vol. 2 (John Wiley & Sons, Chichester, 1992).
(19) J. C. C. Day, Interface Analysis Centre, University of Bristol, UK; http://www.iac.bris.ac.uk.
(20) M. Y. Lin, R. Y. Lo, H. Ogino, and C. Yang, Observation of silicone deposited onto human hair, *Proceedings of the 3rd ASCS Conference, Taipei, Taiwan 1997,* 191–194 (1997).
(21) D. Briggs, in *Practical Surface Analysis, 2nd ed.,* Vol. 2, D. Briggs and M. P. Seah, Eds. (John Wiley & Sons, Chichester, 1992), pp. 367–423.
(22) J. F. Moulder, W. F. Stickle, P. E. Sobol, and K. D. Bomben, *Handbook of X-Ray Photoelectron Spectroscopy,* J. Chastain, Ed. (Perkin-Elmer Corporation, Minnesota, 1992).
(23) J. G. Newman, B. A. Carlson, R. S. Michael, and J. F. Moulder, *Static SIMS Handbook of Polymer Analysis,* T. A. Hohlt, Ed. (Perkin-Elmer Corporation, Minnesota, 1991).

Purchased for the exclusive use of nofirst nolast (unknown)
From: SCC Media Library & Resource Center (library.scconline.org)