

INVESTIGATION OF POSSIBLE CANDIDATES FOR SWIPE SAMPLES IN SAFEGUARDS

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ABSTRACT

Possible candidates for swipes to be used for safeguards are investigated. In connection to nuclear forensics, results obtained from swipe samples at different nuclear facilities are used for attribution involving comparing data and analyses from the swipe to data and analyses from samples of identified sources. Three different candidates

of swipes from commercial on-shelf origins were compared for their applicability. Two of them have chemically modified surfaces to improve their characteristics while the third is an untreated cotton pad. Swipes were investigated in a simulated equivalent environment of nuclear facility. The technique of dust free falling at altitude higher than 15 m was used. Stable isotopes La, As, Zn, Fe, Ni, Cd, Pb, Co, Cr, Cs and Rb in addition to thorium and uranium from reference material were used in the simulation experiment. Surfaces were characterized for their efficiencies using Fourier Transform Infrared spectroscopy (FTIR) and Field Emission Scan Electron Microscope (FE-SEM).

KEYWORDS: Nuclear safeguards, Swipe, isotopes, FE-SEM, FTIR.

INTRODUCTION

Environmental sampling (ES) is a powerful technique used by the International Atomic Energy Agency (IAEA) since 1996. Modern analytical techniques allow for the detection and identification of extremely small traces of nuclear materials. Swipes can provide information regarding the history, origin, processes and type of activities.^[1-3] The analysis of individual particles has advantages compared to “bulk” analysis in which the entire sample is dissolved

and analysed. Particles of man-made materials coming from a nuclear process are normally present as pure compounds, thus greatly reducing the effects of dilution from naturally-occurring elements such as uranium. Particles in the range of several micrometres in diameter can travel large distances from the point of formation, thus increasing the detection probability for samples taken in the general vicinity of a nuclear process. Unlike bulk analysis, which gives only information about the average concentration or isotopic composition in a sample, particles are more representative of the range of elemental or isotopic information present at the inspected location.

The methods for interrogating a surface to detect contamination are as varied as their applications.^[4] The process of interrogating a surface and analyzing the particles collected is widely used in forensics,^[5-9] environmental and regulatory screening.^[10-14] Many agencies have standardized methods for collecting a targeted contaminant and these methods can vary greatly depending on the target particles, the analysis, and the surface itself. A wide range of swipe materials have been used (cotton or rayon gauze pads, microfiber paper, or filter paper),^[4] and in many applications a solvent is applied to the swipe to assist in collection.^[15,17]

Cotton is by far the most popular and universal sampling material. Its drape, tensile strength, and low cost are the most attractive aspects of this fabric. Cotton's relatively low levels of uranium content are another reason that this material (specifically TexWipe TX304) is the current sampler of choice for IAEA environmental sampling use.^[18]

Although muslin-sampling cloths made of cellulosic fibers are effective, some studies have seen them as not ideal sampling materials. Cellulose fibers include a range of chemical moieties that result in heterogeneous binding of analytes. Further, unprocessed muslin sampling swabs contain non-cellulosic compounds found in native cellulosic fibers (i.e. waxes, natural oils and starches) as well as sizing agents and lubricants added during and after textile processing. The decomposition and degradation of an unstable swab material can release contaminants into a detection instrument and therefore interfere with the sample analysis and negatively impact the detection process.^[19]

Further, uranium levels in natural cellulose cannot be expected to remain constant and have been found to vary significantly from batch to batch. To this end, a synthetic material, whose production and uranium levels can be tightly controlled, are likely to be better choice as new

and evolving analytical techniques become more sensitive to the background elements inherent to a sampling matrix.^[18]

Recently, different materials have been evaluated as candidates for surface sampling including, raw glass fibers, polytetrafluoroethylene (PTFE) fibers, and aromatic polyamide polymer fibers. These materials have high thermal stability and are also resistant to mechanical and shredding stresses. However, while PTFE has some suitable properties (e.g., is not wetted by water), PTFE has lower efficiency for collection than muslin or cotton swabs. And, tests on simple glass fiber materials show they do not retain sufficient structural integrity and degrade during use.^[19] A limited number of studies have been reported on swiping efficiency, specifically for nuclear materials,^[20] and these have not attempted to provide a comprehensive evaluation of the factors involved in swiping. These factors include the particle size and composition of the target analyte, the texture and composition of the surface and trap, the force and speed of swiping, and environmental factors that can affect adhesion and removal.

Applying greater force during swiping does not greatly improve collection efficiencies. This fact, coupled with the observation that many particles are detached but not collected, implies that improvements in collection efficiency are dependent on improvements in adhesion of the particles to the collection surface, rather than larger forces to detach the particles.^[21]

This study investigates new swipe candidates for their ability to be stable, enhance sensitivity for particle level detection, and enhance analyte adsorption and collection through their surface-active groups. Polymeric-based fabrics are particularly attractive because a wide range of commercial materials exist, the materials can be engineered into the desired form, with very low-background materials. The selected baseline for comparison was the IAEA's standard muslin sampling material (TX304).

MATERIAL AND METHODS

The engineered experimental approach was designed to simulate real swiping of equivalent environment to nuclear facility. In an attempt to control the experimental factors affecting the efficiency of swiping, a flat glass surface with dimension of 26 cm x 18 cm was set into quarter with a concentric circle of 12.5 cm diameter as shown in Fig.1.

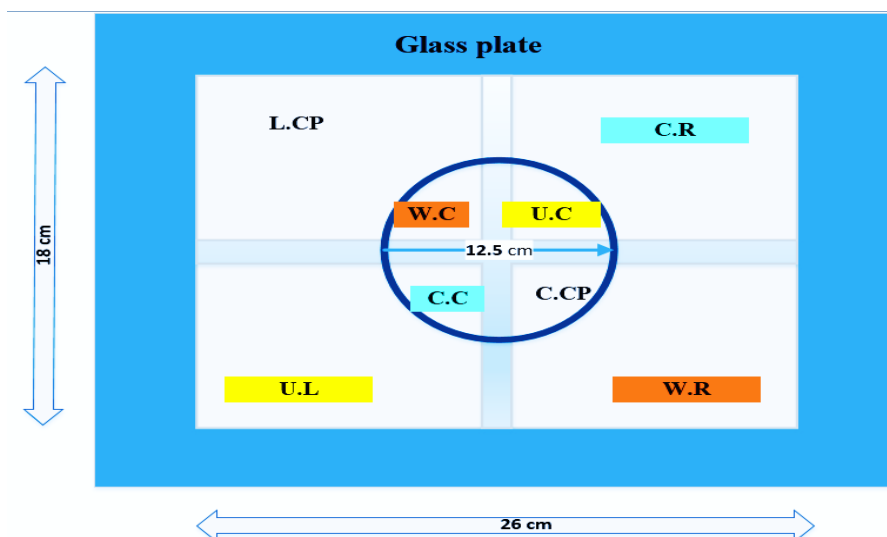


Fig. 1: Diagram of sprayed glass area, C.C, W.C, U.C and C.CP are central swiping positions of the tested swipes while C.R, W.R, U.L and L.CP are the corresponding peripheral swiping positions.

The technique of dust free falling at altitude higher than 15 m was used to limit the particle size of falling dust within 5~10 μm . To achieve a homogeneous surface distribution as feasible, spraying was done from left to right, top to bottom and opposite direction while swiping along the stream of the spray as illustrated in Fig.1. Standard solution (1000 ppm) of fourteen stable isotopes La, As, Zn, Fe, Ni, Cd, Pb, Co, Cr, Na, Cs and Rb in addition to Th and U from reference material were mixed in analytical grade ethanol and sprayed to the investigated surface. These elements were selected as being possible fission and activation products which may exist in hot cells of research reactors.

The surface was sprayed and allowed to dry after each spray round till the whole solution was consumed. Swiping was done from the peripheries and the center to investigate the impact of spraying on particle distribution. When swiping, particles are removed primarily by three mechanisms, *lifting, rolling and sliding*.^[21] For the purpose of environmental sampling for safeguards, the interest is focused on lifting, which is resulting from the attachment of the particles to swiping material. Lifting occurs when the forces attaching the particles to the swipe exceed those adhering the particles to the swiped surface. Three different sampling materials were selected for evaluation. Two commercial tissues treated with surfactant and plain cotton pads were used in comparison to the cotton-based TexWipe TX304, which was used as a standard and baseline for comparative evaluation. Fig. 2 presents images of the native swipes used in the study.

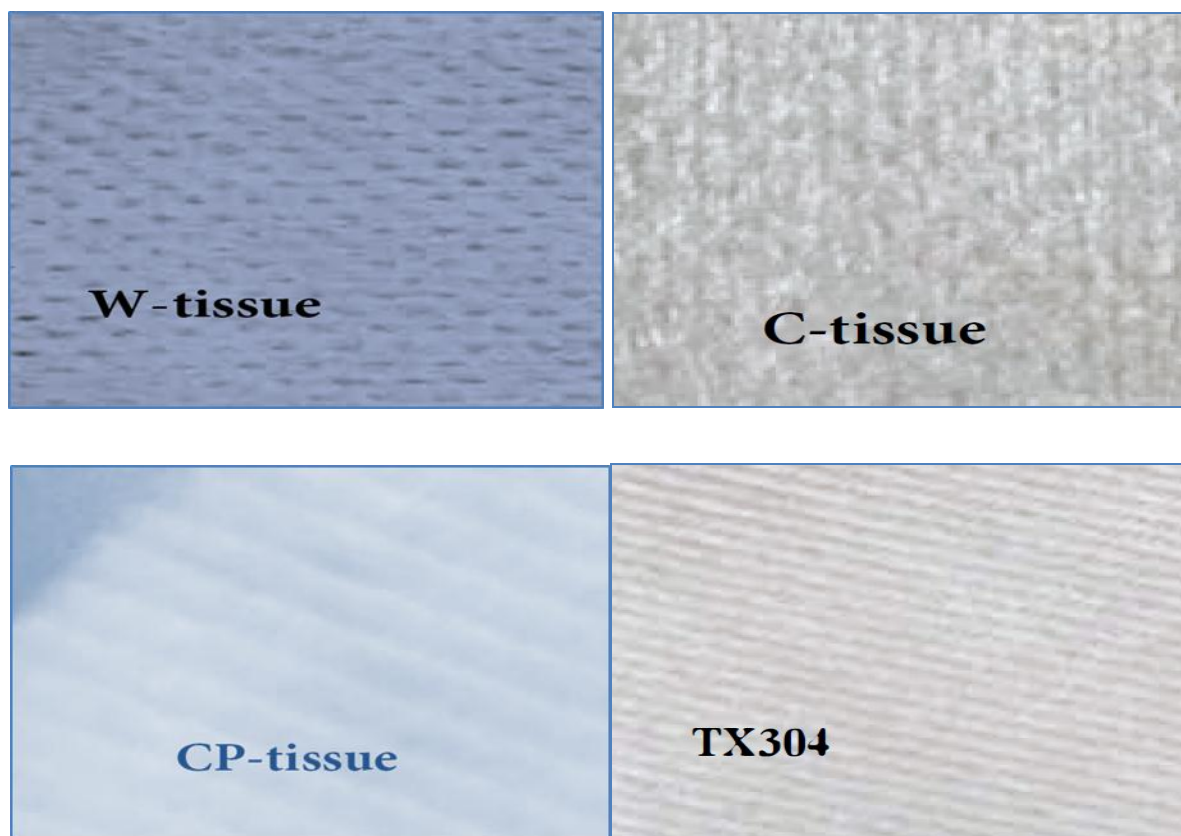


Fig. 2: Samples of the standard and tested environmental sampling materials. Swipe W- and C-tissue are the treated tissues; CP is the cotton pad and the cotton-based TexWipe TX304 standard material.

The used commercial tissues are laundry sheets that usually comprising support matrix, colour catcher and fabric softener. The support matrix can be any type of natural or synthetic material with which a colour catcher may become associated, if the material used is capable of either delivering the mobile colour catcher to the washing liquor, or retaining some association with the stationary colour catcher, or both. Further to its function as a carrier for the stationary colour catcher, the purpose for the support matrix is to provide a sufficient surface area upon which the stationary colour catcher is accessible to wash liquor. Materials, which are suitable for support matrices, include both cellulosic and non-cellulosic fibres in both woven and non-woven form. Preferably, the support matrix comprises substances that have absorptive capacity and/or contain reactive groups. Cellulosic such as wood pulp, rayon and cotton are especially effective substances, besides having the additional advantage that they are available at relatively low cost. Synthetic polymeric materials such as polyester, polyethylene and polypropylene may also be used as support matrices alone, or in

combination with other support matrices as additives to improve fabric wash strength under standard washing conditions.

Analysis of swipes

The Scanning Electron Microscope (SEM) used for investigating the swipes is Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., magnification 14x up to 1000000 and resolution for Gun.1n), FEI company, Netherlands. SEM relies on the production of a micro-focused electron beam, which is then scanned over the surface of the specimen in vacuum. When this beam of electrons strikes the sample, scattering of the beam by the surface is used to make a magnified image of the sample surface. While elastic scattering of the beam at high angles of reflection gives image in which is sensitive to high atomic weight elements (backscattered electron mode), X-ray emission by atoms in the sample gives an elemental map of the surface or the elemental composition of a single spot on the sample surface (X-ray fluorescence mode). The detection of fluorescent X-rays coming from the sample can be accomplished with a solid-state detector which measures X-rays of a wide range of energies (energy-dispersive or EDX detection). The study was focused on selected particles over several mm² of the investigated surface using first the backscattered electron signal to locate the glowing heavy particles, then the EDX system to measure the spectrum of each particle found.

For surface characterization and identification of inorganic minerals and function groups, a Bruker VERTEX 70v FTIR Spectrometer was used. This FTIR spectrometer technology is capable of covering the FIR/THz and MIR in a single scan. The basic principle of the FTIR technique is based on the fact that within any molecule/polymer the atoms or groups of atoms vibrate with a few definite, sharply defined frequencies characteristic of the compound. The IR frequencies are indicative of the transitions between vibrational energy states for specific bonds and types of vibrations and lie in the infrared region of the electromagnetic spectrum. When the sample is placed in the IR beam it absorbs energy at specific frequencies characteristic of the molecule and transmits all other frequencies. The resulting pattern is known as a molecular infrared vibrational absorption spectrum. The intensity of the absorption is a known reproducible function of the amount present.

RESULTS AND DISCUSSION

Effect of swipe material

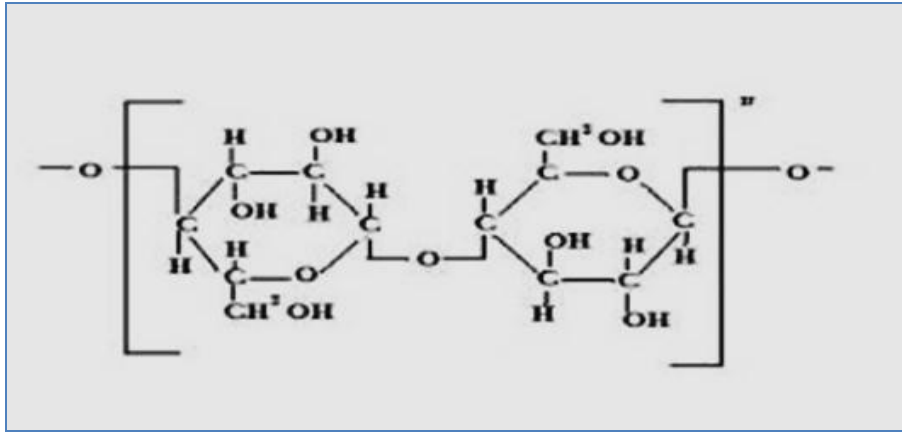
As discussed before, swipes may be natural or synthetic fibers or a mixture of both. Literature defines suitable materials as poly-olefins, poly-(haloolefins), insoluble poly-(vinyl alcohol), polyesters, polyamides, poly-acrylics, protein fibers and cellulosic fibers. Both laundry tissues textile are having a functionality dye complexing moieties bound to the textile article and a water-soluble container enclosing a composition mint for other functionality as a bleach booster or complexing agent. Side chains are grafted onto the textile to provide the desired functionality.

Each of the investigated native tissues was imaged by FTIR. Fig 3 presents the combined spectra to illustrate differences between the investigated tissues and the baseline TX304. Table 1, presents the elemental analysis of swipes, while Fig 4 (a-d) presents the spectra obtained by FE-SEM.

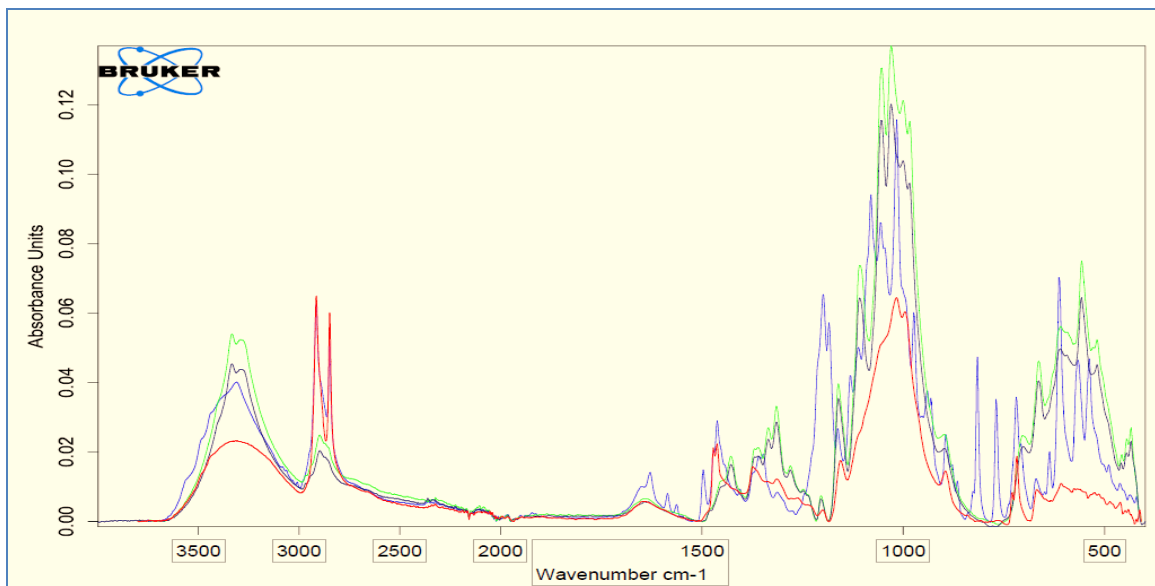
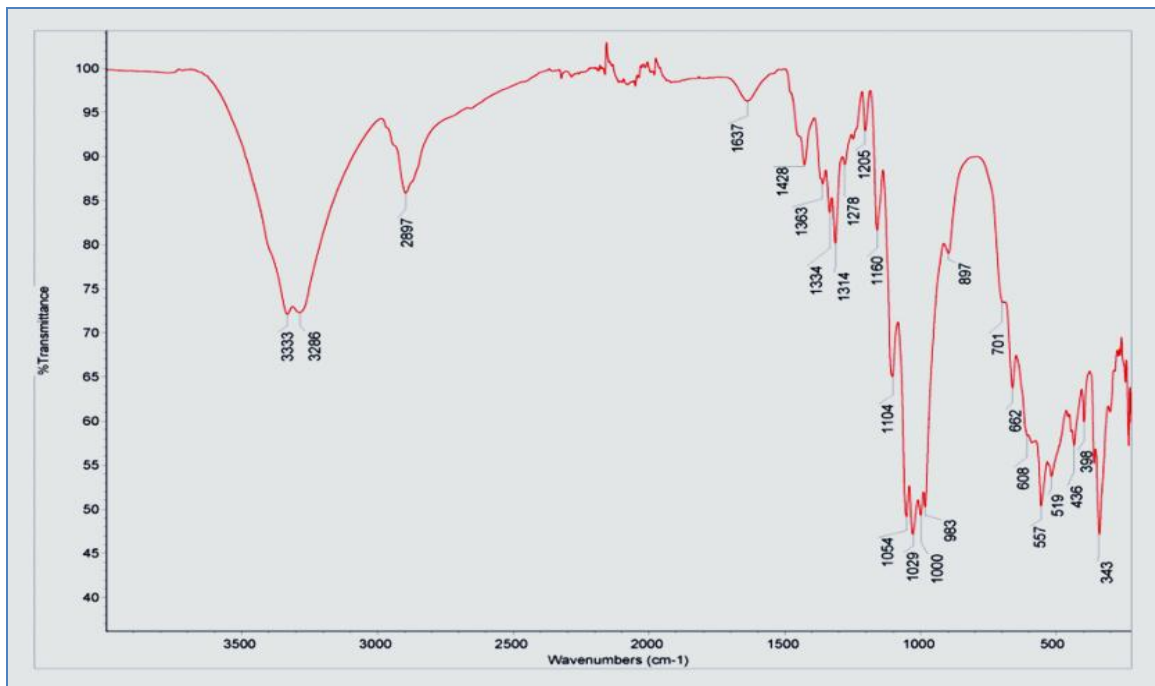
Table 1: SEM Elemental analysis of the raw tissues given in (%).

Element	W tissue	C tissue	TX304	CP tissue
C K	60.23	65.60	47.17	48.74
O K	27.00	32.21	51.60	51.26
NaK	5.04			
S K	5.64	0.52		
ClK	1.14			
FeK	0.94	1.38	0.83	
SiK		0.29	0.39	

Both muslin “TX304” and the plain cotton pad “CP tissue” gave similar elemental values (in %) for C and O and FTIR spectra of cellulosic fibers as the main component of their material. Traces of Fe and Si in both TX304 and C tissue are most likely due to contamination of fine dust during handling. Cotton fiber is made of a long molecular chain of cellobiose and contains hydroxyl O-H in their molecular structure. The FTIR characterization of the cotton fiber confirmed the high cellulose and low hemicellulose/lignin content (see Fig 3) as reported in the literature.^[22-24] In both laundry tissues, C and W, the FTIR spectrum of polyethylene (PE) is clearly identified. The characteristic PE absorbance bands are located at 2914 cm^{-1} , 2847 cm^{-1} , 1470 cm^{-1} and 718 cm^{-1} . This indicates that the supporting matrix contains synthetic polymeric materials, in this case polyester, in combination with cellulosic material such as wood pulp, rayon or cotton.



Cotton structure and FT-IR spectrum.^[25]



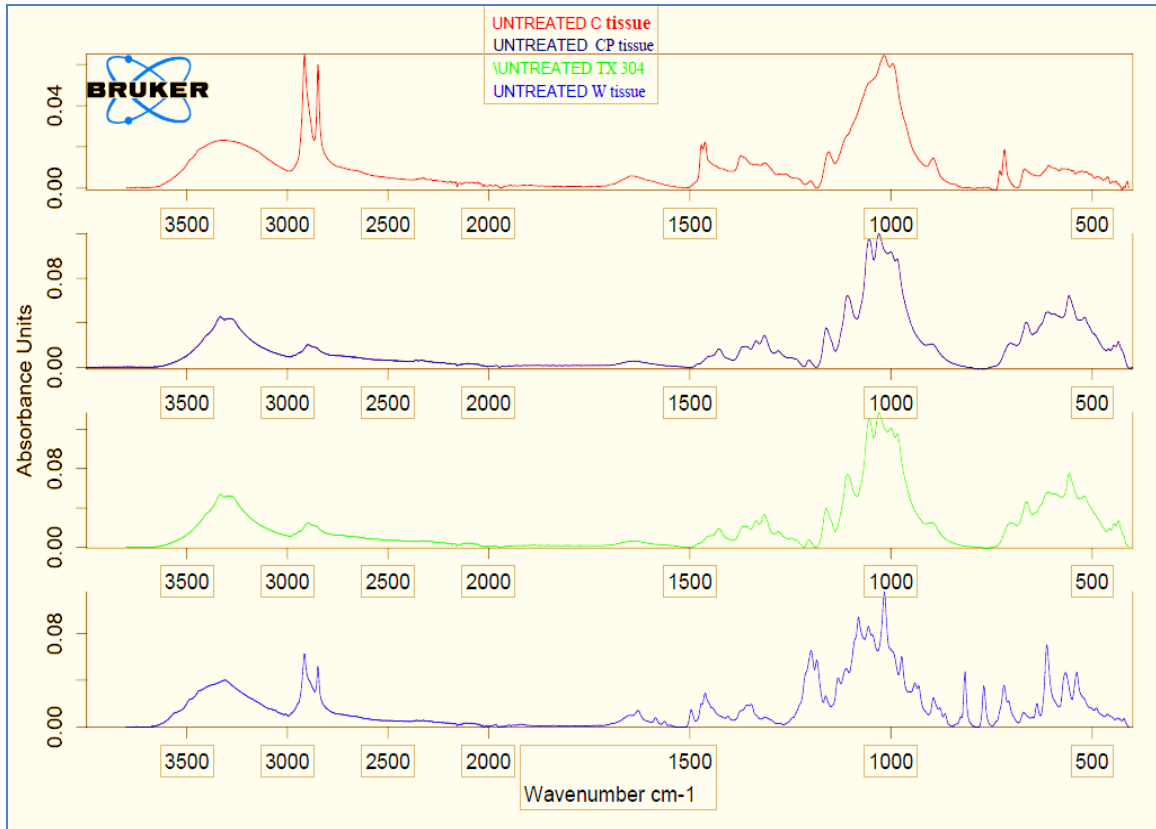


Fig. 3: Combined IR spectrum of investigated tissues; C tissue, W tissue, CP and the reference TX30.

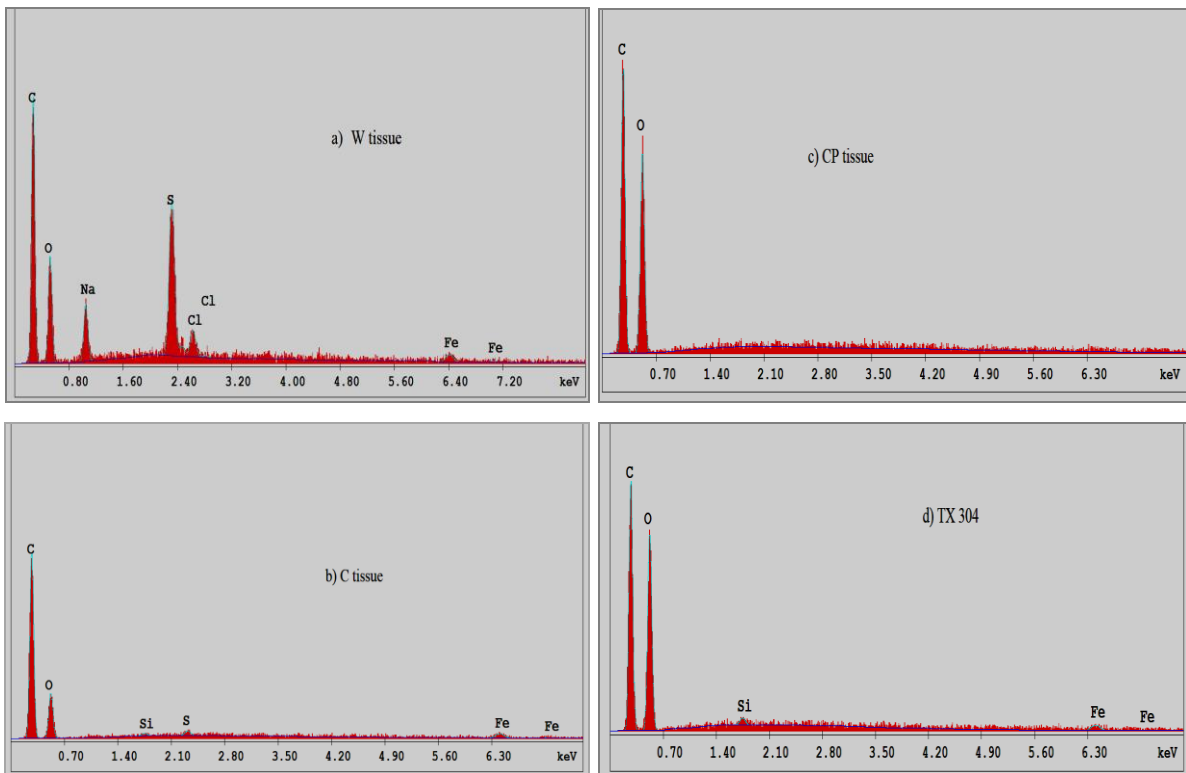


Fig. 4: FE-EDX Elemental analysis of C tissue, W tissue, CP and TX304.

Surface images made by SEM for native “W tissue” textile showed that the tissue is impregnated and have deposited therein the composition for the desired functionality (see Fig 5 a). The component is probably a complexing agent able to bind metal ions. The composition may, for example, contain surface-active agents such as an anionic, non-ionic, cationic, amphoteric or zwitterionic surface-active agents or mixtures thereof.

In our FTIR interpretation, we focused on the well-characterized regions between 3500 to 2800 cm^{-1} and 1800 to 1550 cm^{-1} . Additional changes in absorption bands below 1500 cm^{-1} were found to be more difficult to associate with specific functional groups. The vibrational modes in the 3500 to 2800 cm^{-1} region are mainly due to stretching the O-H and C-H.^[22,23] The modes in the 1800 to 1500 cm^{-1} region are from the carbonyl functional group C=O, as well as some overlapping involvements of C-H bending vibration. The presence of a carbonyl/carboxyl functional group is indicated by the presence of a strong band around 1650 to 1780 cm^{-1} .^[24]

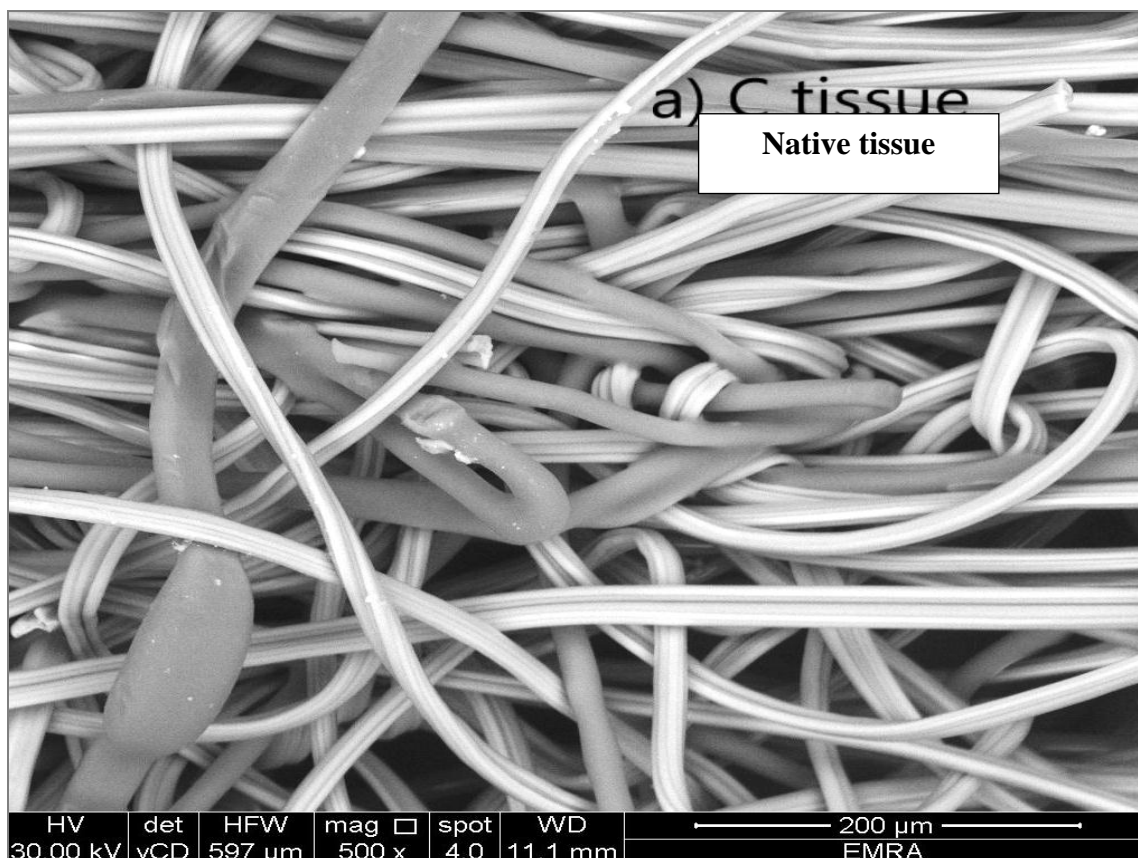
The W tissue showed strong C-Cl stretching at 850-550 cm^{-1} indicating halo compound. The strong sulfonate S=O stretching at 1372-1335 is also identified. FE-EDX analysis of W tissue showed Na, S, and Cl, which support the IR results proposing the presence of alkali metal (probably sodium) aluminosilicates and Alkyl sulfate.

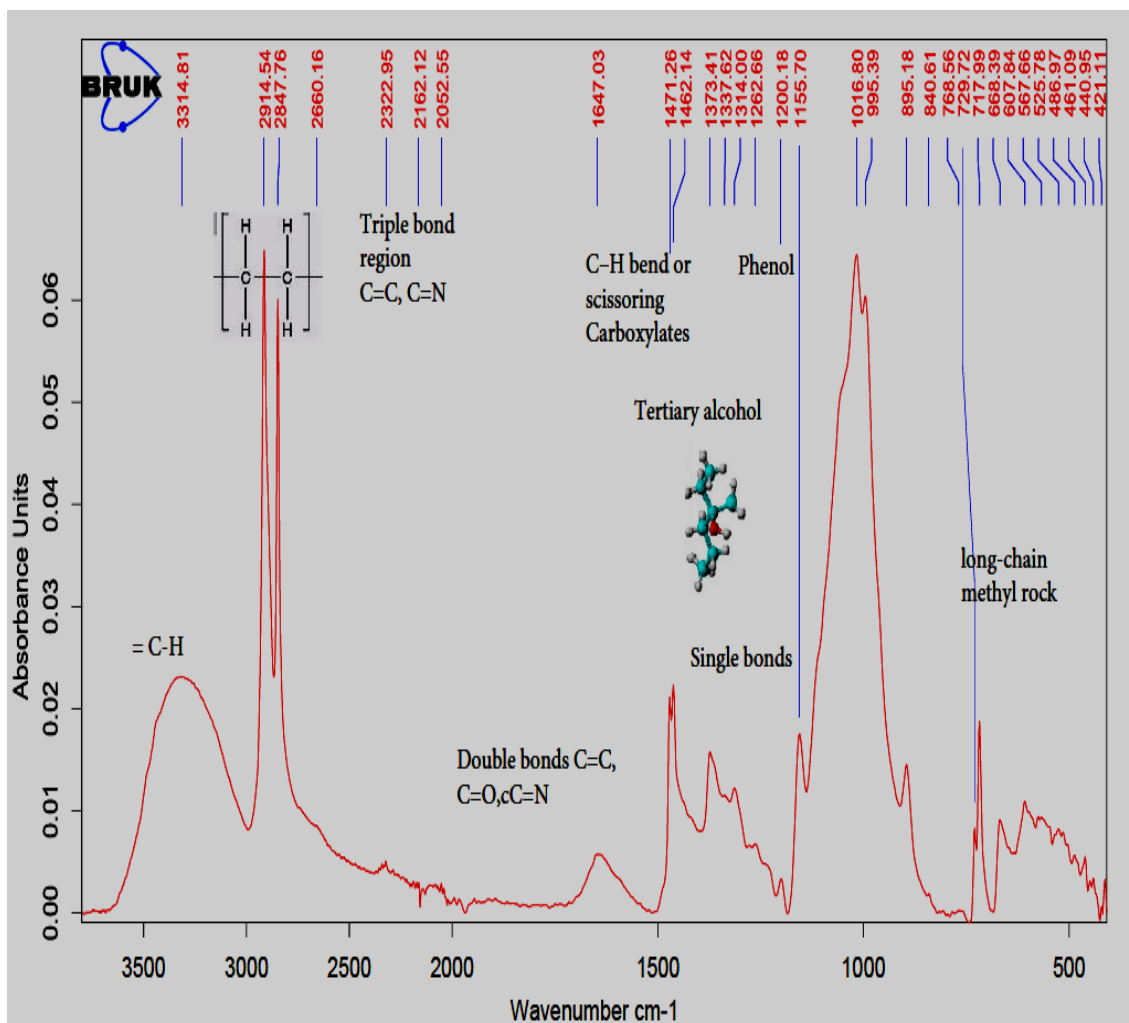
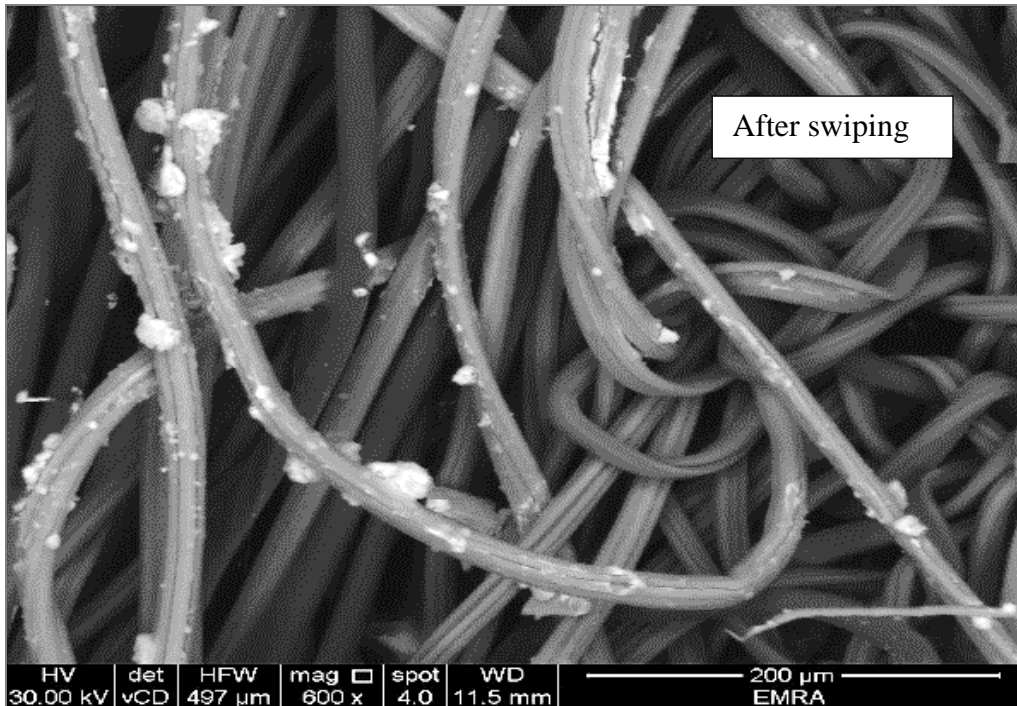
In Fig 5 a), the FTIR spectra for C tissue before and after swiping showed strong peak at 1325 cm^{-1} , the CH_2 wagging vibration in cellulose. The absorbance of the peak after swiping increased from about 0.02 to 0.3 absorbance unit. The peak at 1647 is shifted to 1638 of the C=O stretching vibration in conjugated carbonyl. The peak at 3300 is shifted to 3339 cm^{-1} with pronounced increase in absorbance from 0.04 to 0.17 proposing complexation with the OH group. *Although applying greater force* during swiping does not greatly improve collection efficiencies, it is believed to be the reason behind the increase in absorption. Most likely, it did increase the number of function groups compressed in the small scanned area.

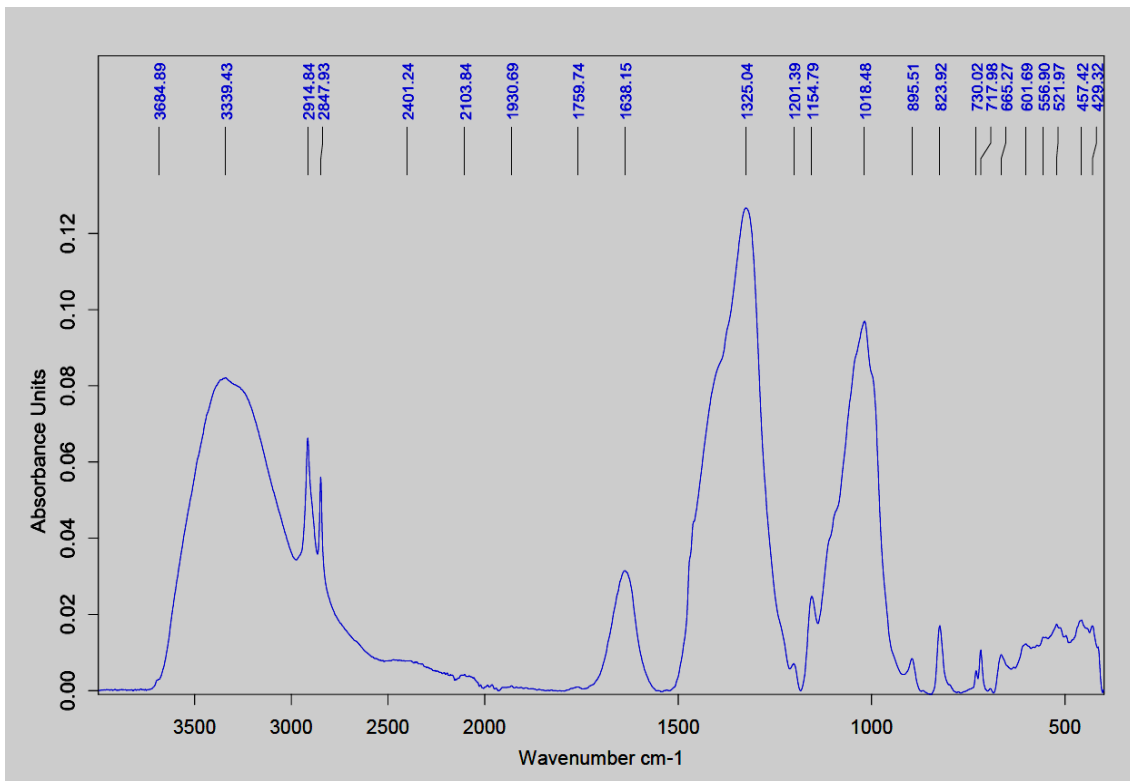
Fig 5 b) presents the FTIR spectra of W tissue before and after swiping. The peak at 3314 is shifted to 3339 cm^{-1} with increase in absorbance from about 0.01 to 0.10 absorbance unit proposing complexation with the OH group. The peak at 995 cm^{-1} , is shifted to 1325 probably due to bending vibration of the OH group and the absorbance of the peak after swiping increased from about 0.02 to 0.11 absorbance unit.

These results propose that there is a mechanism of interaction between the function groups on the surface of both laundry tissues and the swiped particles. It is likely that the combination of synthetic polymeric materials, in this case polyester, and cellulosic material improved the characteristics of the tissue. It is likely that, due to air humidity, the water-soluble container grafted onto the textile enclosing the composition mint for complexing agent were partially dissolved and enhanced the interaction between swiped particles and the surface.

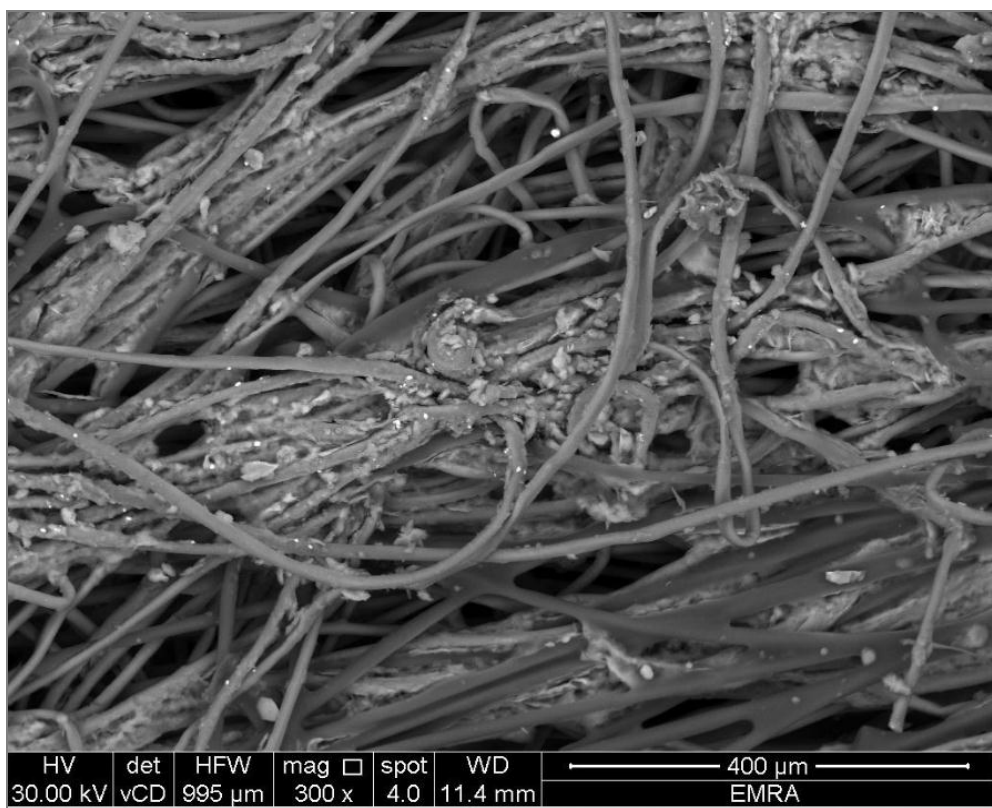
Images of SEM presented in Fig 5 c) propose that the main mechanism of particle collection in case of the standard swipe tissue TX 304 is simply holding of the swiped particles within the roughness of the fiber structure of the textile surface. Similarly, does the commercially on-shelf available cotton pad (graph d). However, results of FTIR shows slight shift in the peak 3291.18 and 3389.18 to 3282.68 and 3335.9 cm^{-1} respectively. This propose complexation with the OH group. The peak at 1639 is shifted to 1635 cm^{-1} , which propose complexation with the C=O group.

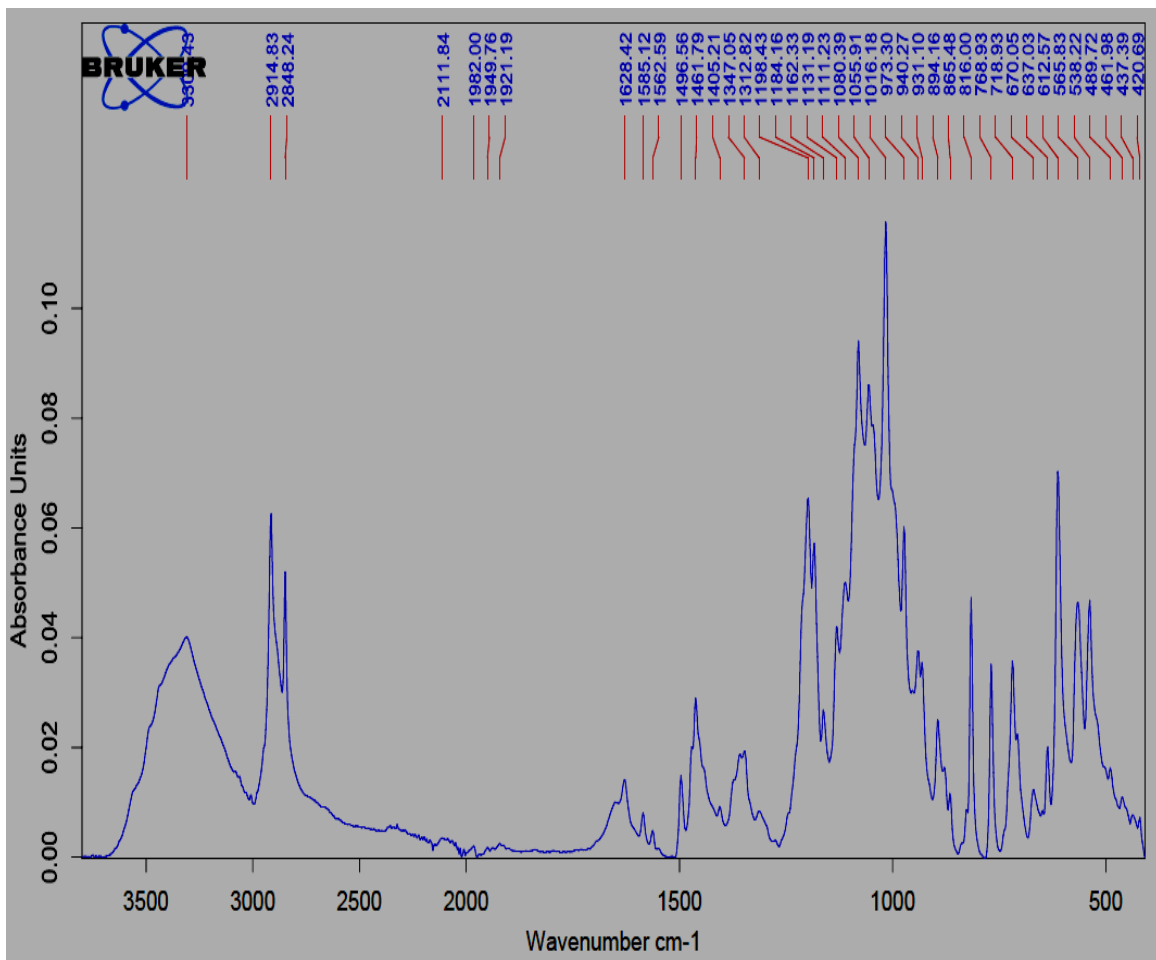
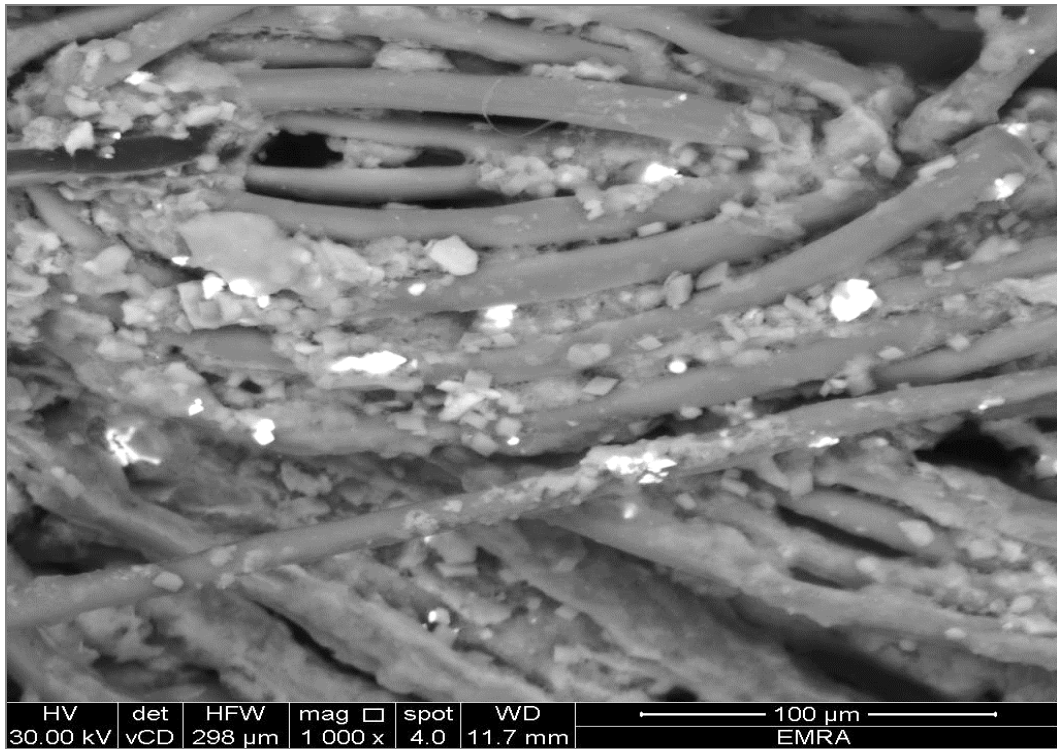


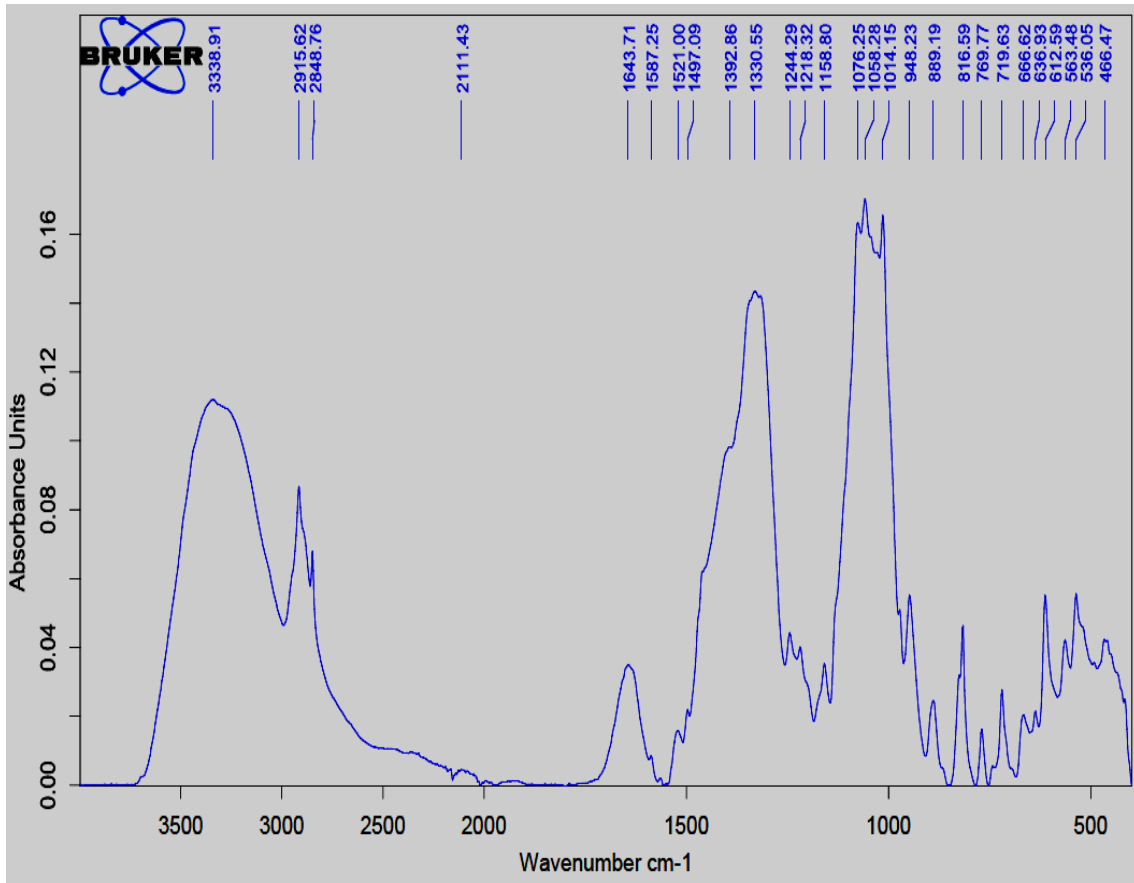




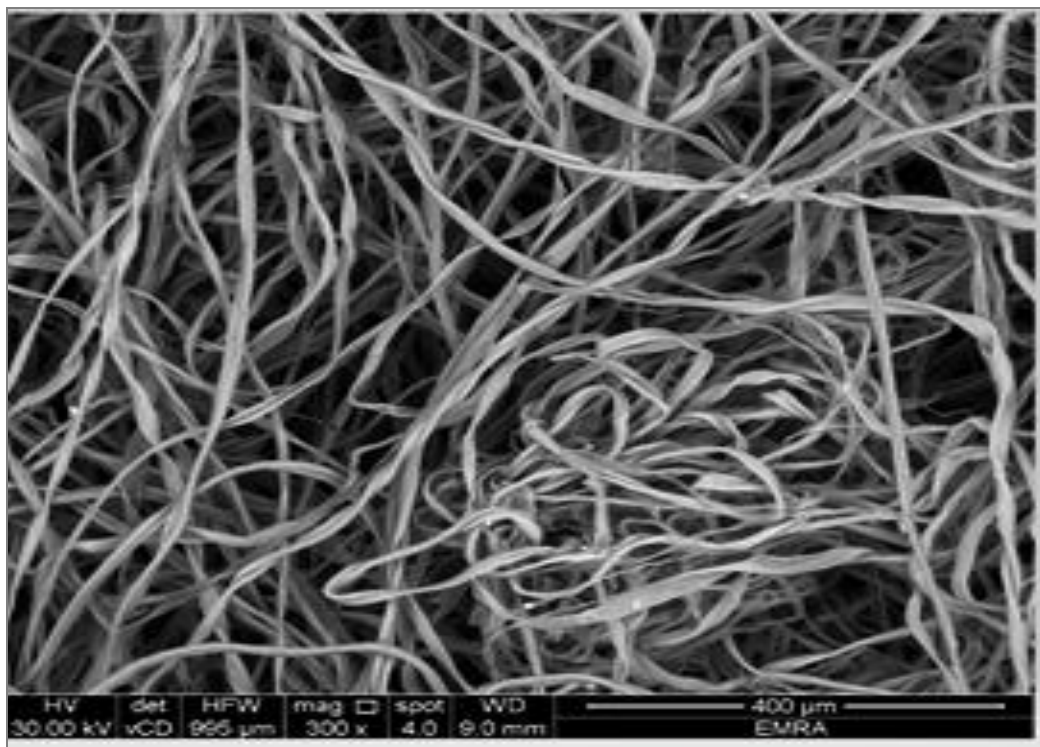
a) FE-SEM images and FTIR spectra of 'C tissue" before and after swiping.

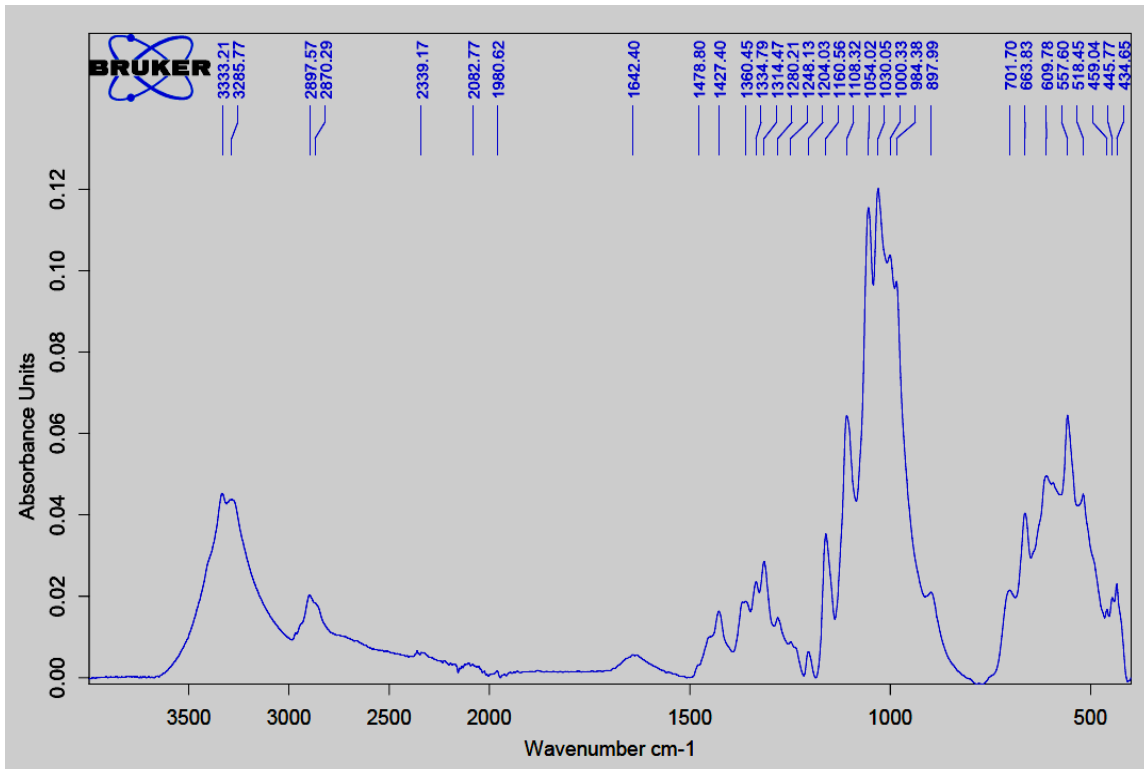
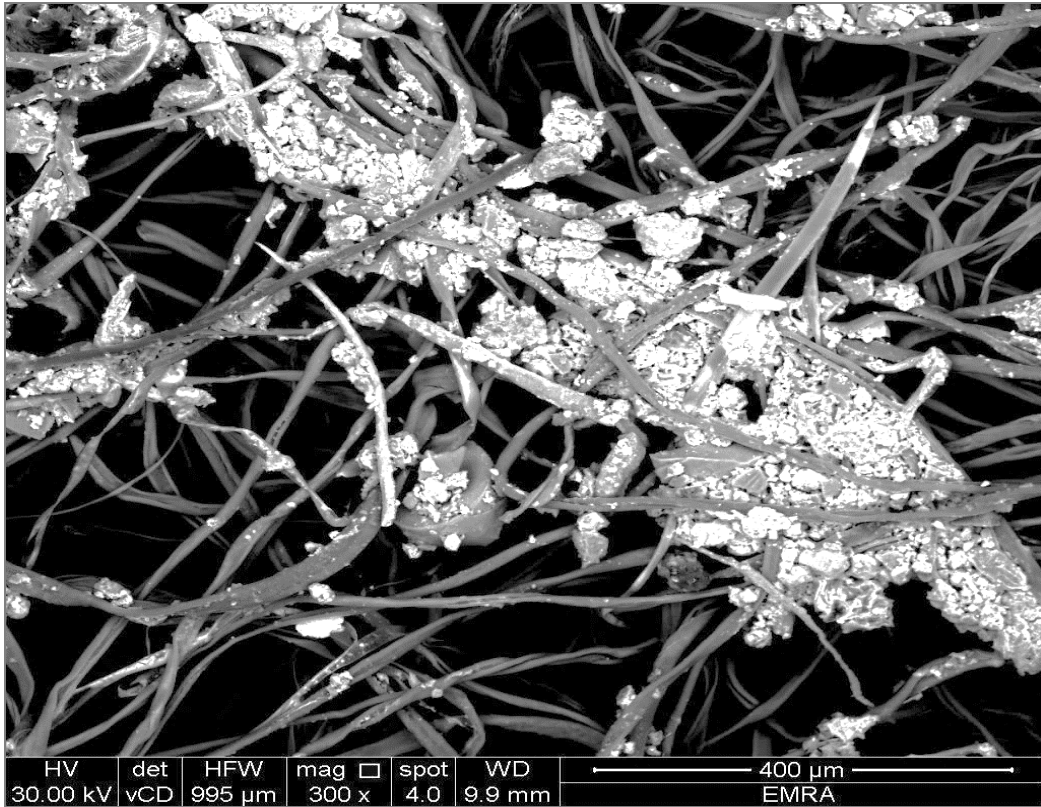


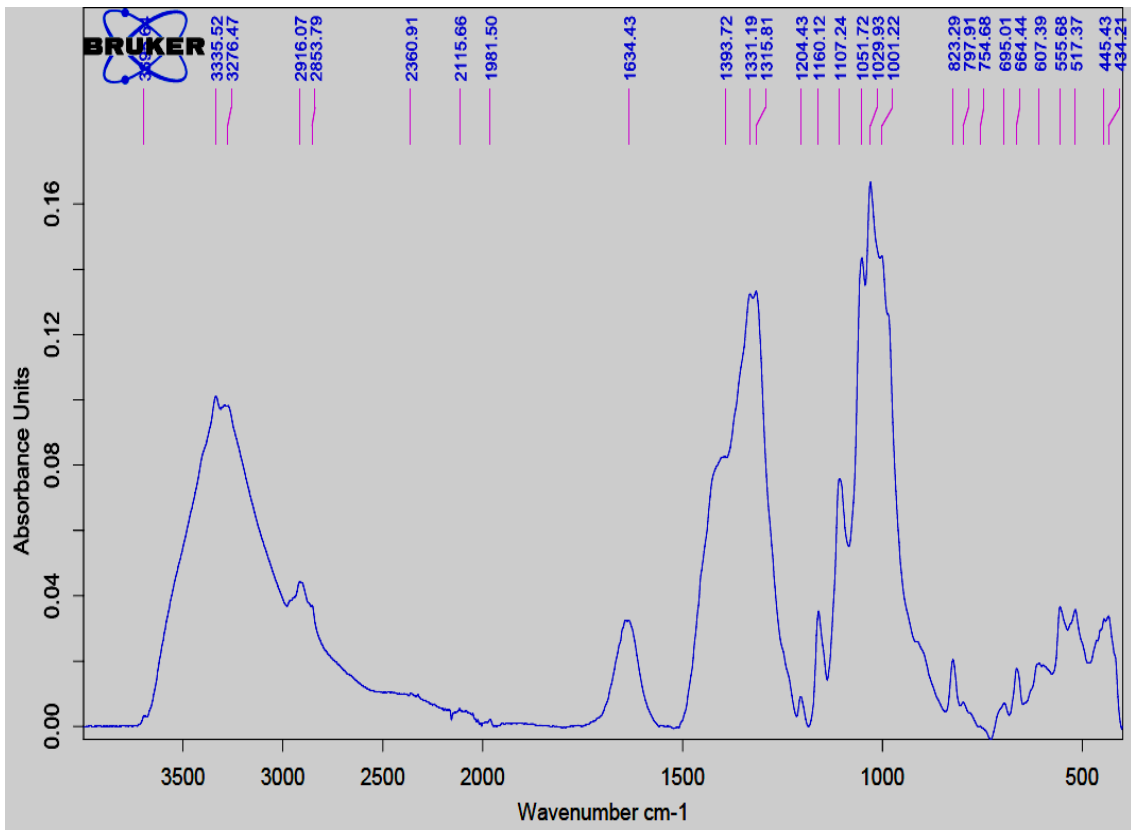




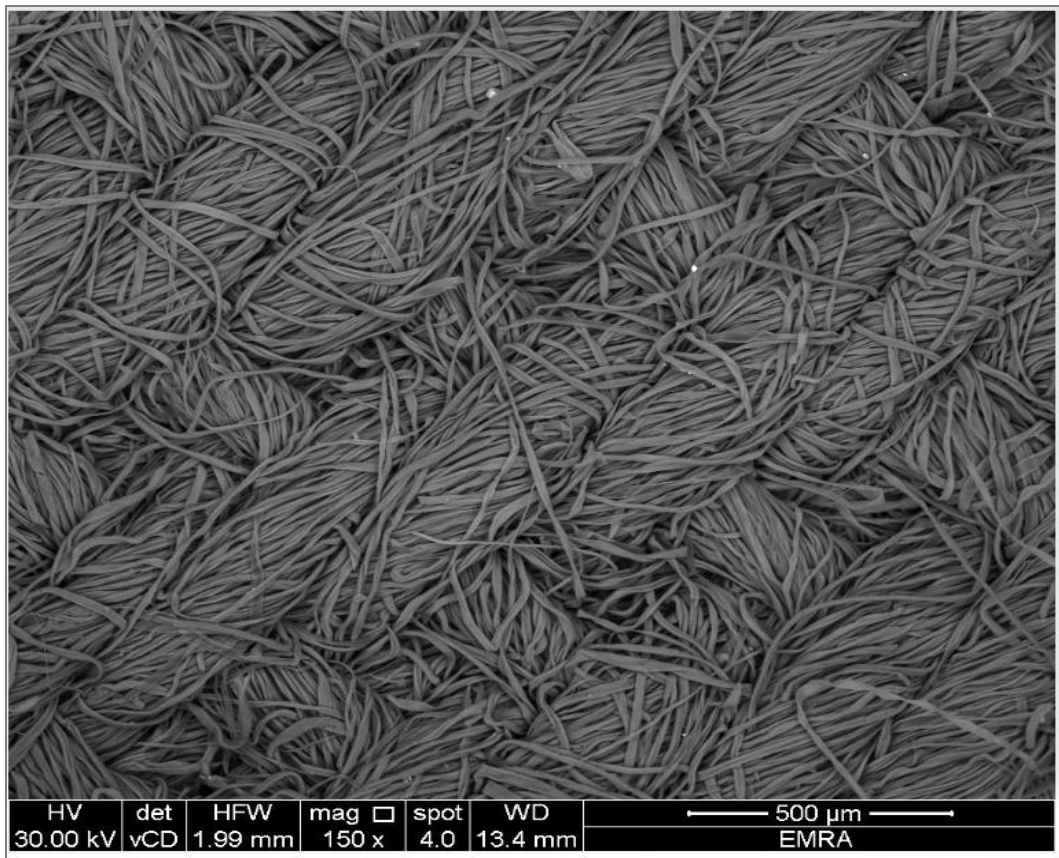
b) FE-SEM images and IR spectra of W tissue before and after swiping

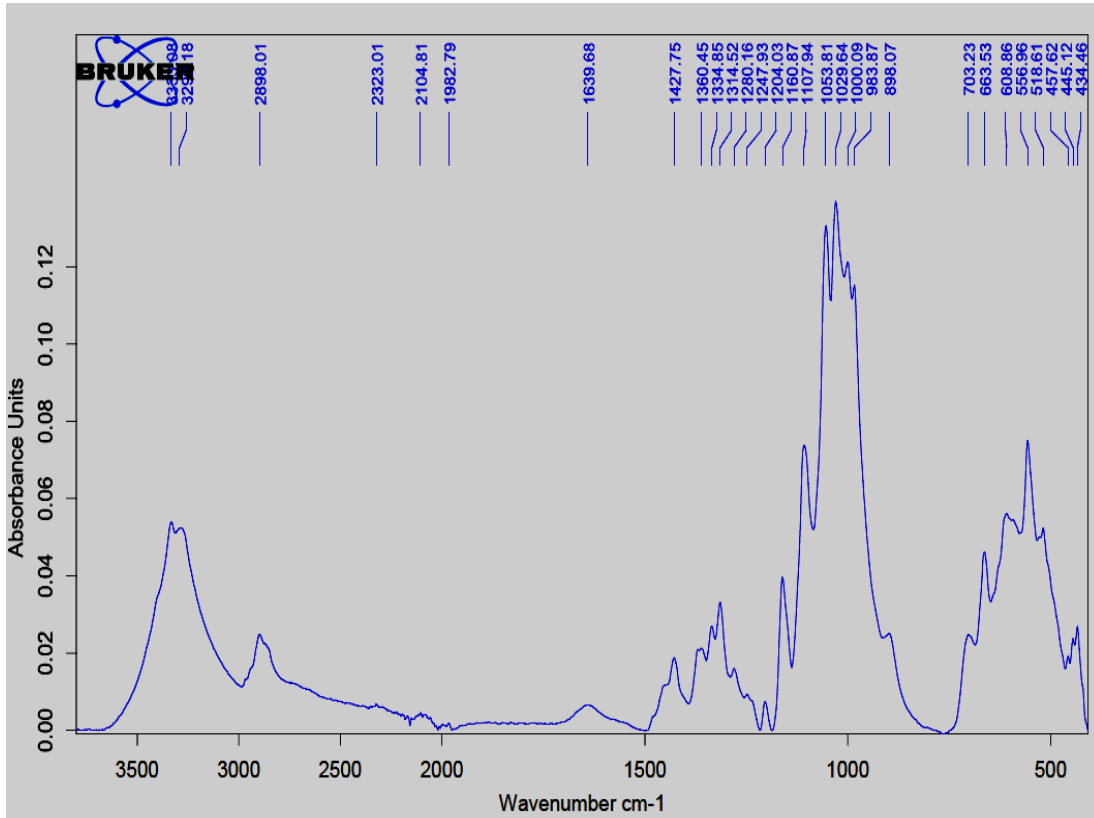
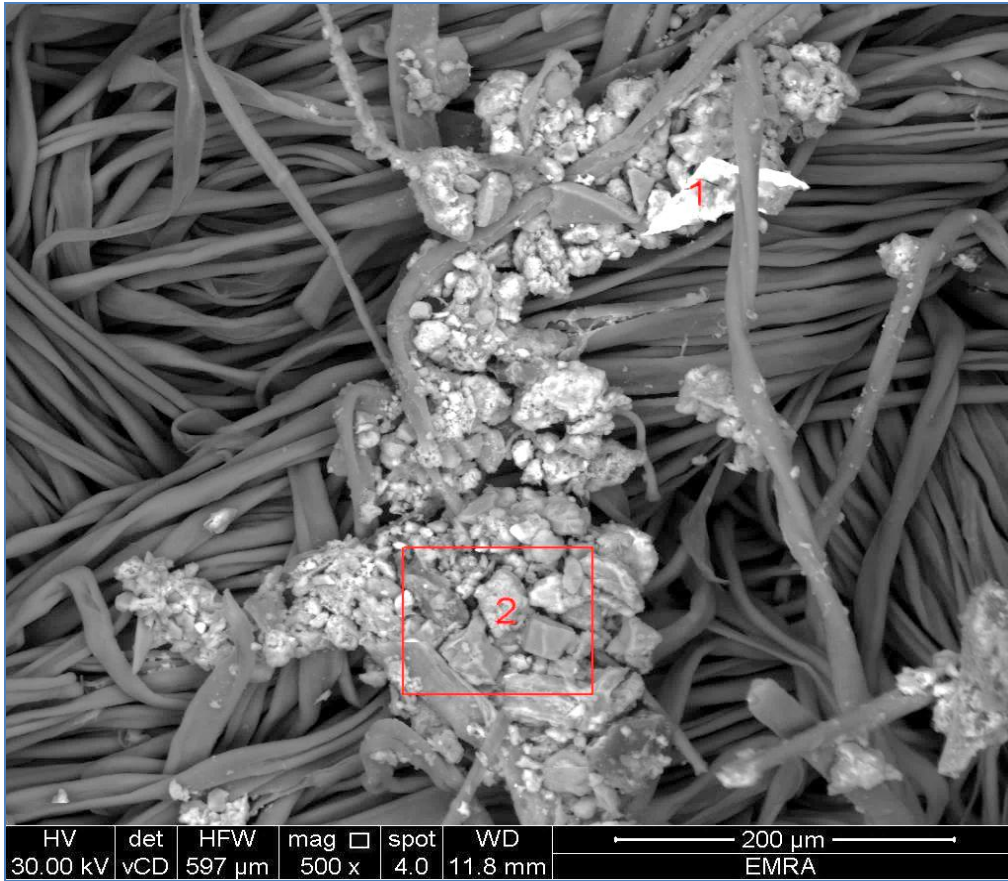


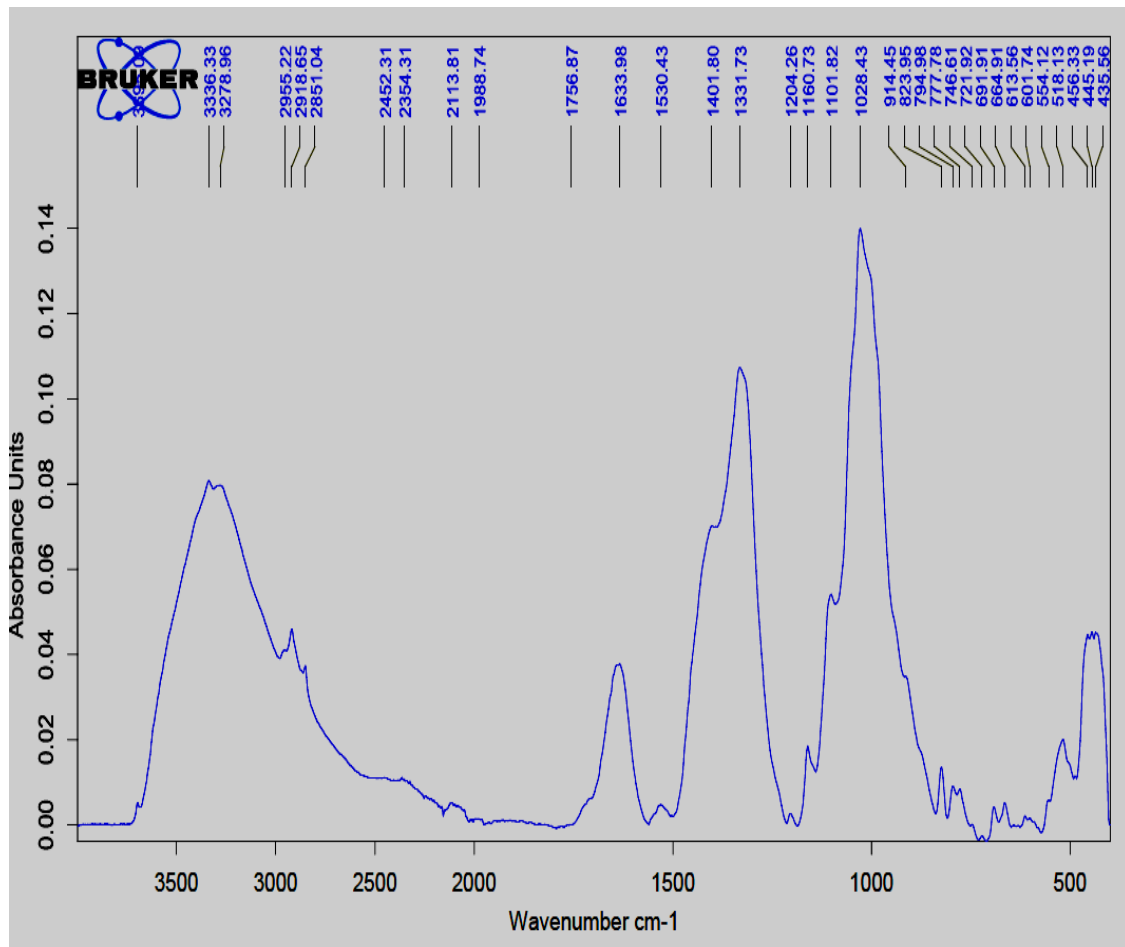




c) FE-SEM images and IR spectra of CP tissue before and after swiping.







d) FE-SEM images and IR spectra of TX 304 standard tissue before and after swiping
 Fig 5: FE-SEM images and IR spectra of investigated swipes; a) C tissue, b) W tissue, c) cotton pad and d) Muslin TX304.

Effect of spraying

Swipes were collected from the central circle area and the peripheral rectangular area of the sprayed surface as presented in Fig 1. Each of the swiped areas in the center, which presents a quarter of circle, was almost 30% of the rectangular swiped area. Fig. 6 (a-d) presents the weight (%) of elements normalized to their swiped areas for the investigated 4 swipes. Increasing the swiped area was not necessarily adding value to the effectiveness of swiping. In most of the cases, swipes taken from the center were more informative than those taken from the peripheries were. This could be due to the mechanism of spraying, the spraying solid angle, and the load of particles to the surface and the directional force of collection as well. Fig 7 presents the results of FE-SEM in weight present for elements used to simulate the swiped environment in the two positions, peripheries and center among the swiping candidates.

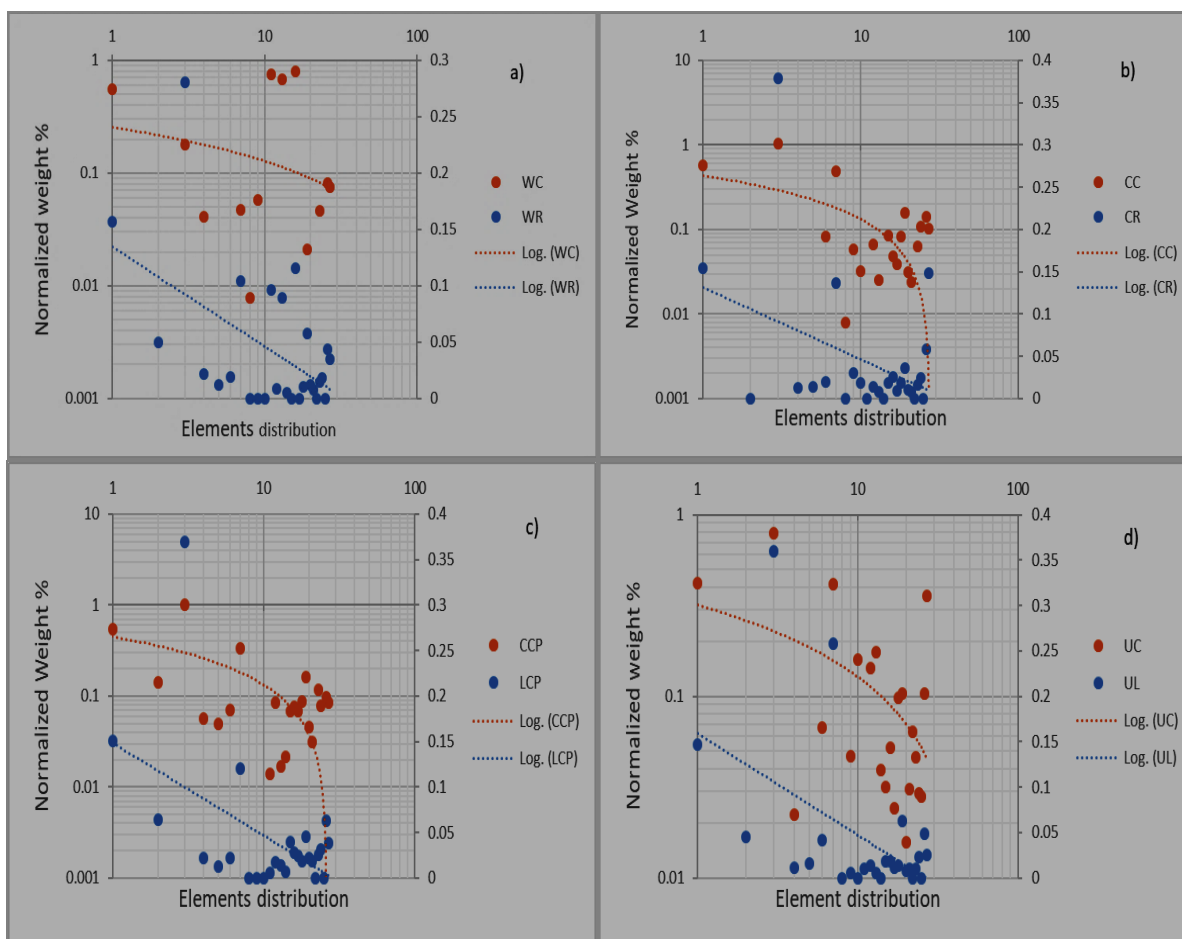


Fig. 6 (a-d): presents the weight (%) of elements normalized to their swiped areas for the investigated 4 swipes.

Bearing in mind that the study is qualitative rather than quantitative, attention was paid to both the ability of the tissue to accumulate particles with the largest number of investigated elements and the ability to retain the highest concentration of these elements. The C tissue showed representative swipe with the highest content of La, Zn, U, As and Rb. The cotton-based polyester-containing matrix provided for more active groups that improved the collection efficiency of the swipe. It is believed that the reason for this is the combination of C=O in conjugated carbonyl/carboxyl, the O-H and C-H group, and the OH group. The W tissue showed the highest content of light elements such as Cs, Cd and Fe, even though heavier elements such as Pb and La were not detected. This could be due to the interaction of Cs and Cd with the C-Cl group, which showed strong stretching at $850\text{-}550\text{ cm}^{-1}$. The displacement of the alkyl metal on the surface and interaction with the sulfonate S=O could be the main mechanism of retaining these elements.

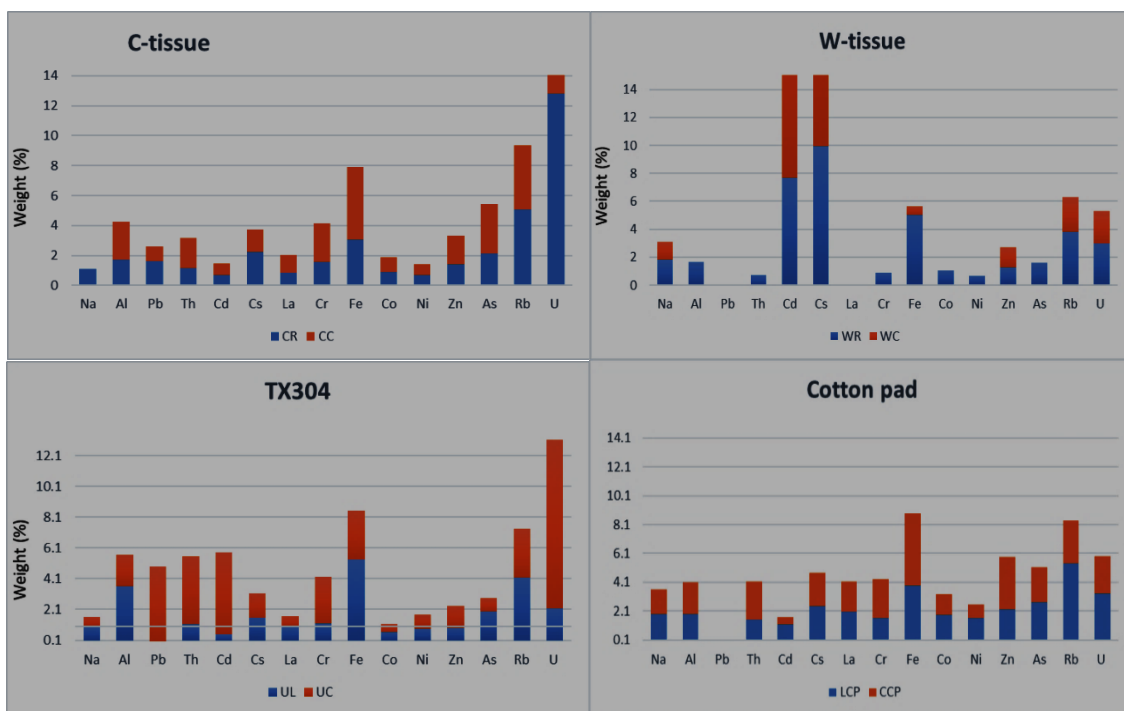


Fig 7: Distribution pattern of the elements used to simulate the swiping experiment where WR is W tissue peripheries, WC is W tissue center, CR is C tissue peripheries, CC is C tissue center, LCP is cotton pad peripheries, CCP is cotton pad center, UL is TX304 peripheries and UC is TX304 center.

Bearing in mind that the study is qualitative rather than quantitative, attention was paid to both the ability of the tissue to accumulate particles with the largest number of investigated elements and the ability to retain the highest concentration of these elements. The C tissue showed representative swipe with the highest content of La, Zn, U, As and Rb. The cotton-based polyester-containing matrix provided for more active groups that improved the collection efficiency of the swipe. It is believed that the reason for this is the combination of C=O in conjugated carbonyl/carboxyl, the O-H and C-H group, and the OH group. The W tissue showed the highest content of light elements such as Cs, Cd and Fe, even though heavier elements such as Pb and La were not detected. This could be due to the interaction of Cs and Cd with the C-Cl group, which showed strong stretching at $850\text{-}550\text{ cm}^{-1}$. The displacement of the alkyl metal on the surface and interaction with the sulfonate S=O could be the main mechanism of retaining these elements.

TX304 and cotton pad showed relatively fair distribution of all elements except Pb with quite large difference between swipes taken from center and peripheries. This could be due to the looseness of particles on the surface of the tissue as observed during SEM investigations.

This suggests that the binding to the surface is in favor adhesion force than chemical interaction.

Additionally, the results of FE-SEM showed the presence of additional elements, which are due to environmental pollution and contribution of dust. Fig.8 presents the distribution pattern of all detected elements in the four investigated swipe. The C tissue presents a well-balanced fingerprint distribution pattern for all elements.

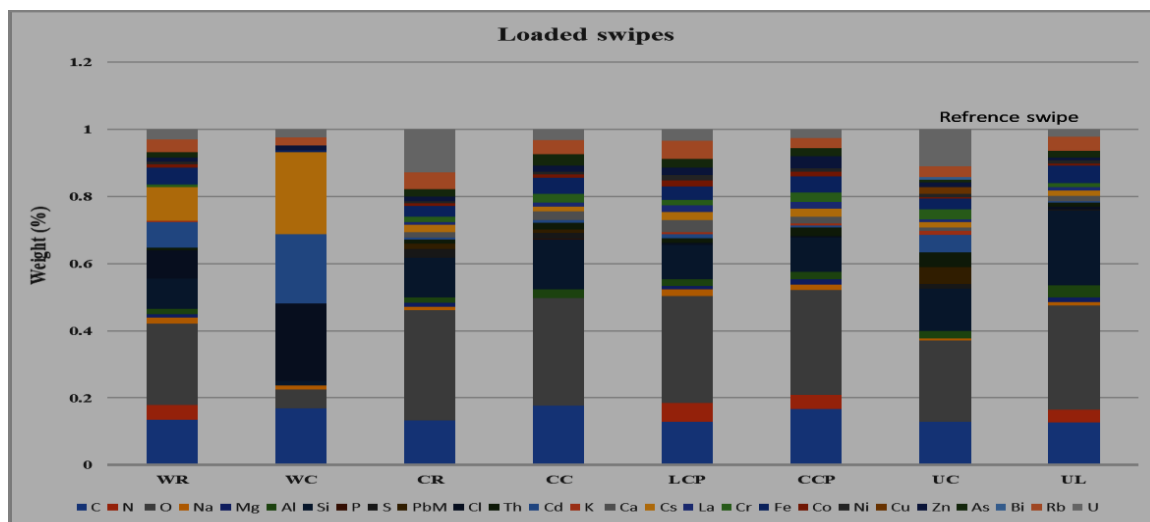


Fig. 8: Results of FE-EDX elemental analysis of C tissue, W tissue, CP and TX304.

CONCLUSION

Results of engineered experiment concluded that the surfactant treated C tissue presents powerful candidate for environmental swipe samples. The design performance of the chosen sampling methodology (lifting), and the directional force applied to the exposed surface area have strongly improved the effectiveness of collection, and increased the efficiency due to better adhesion of fine dust particles to the textile of the swiping material specifically at the wide end of spraying solid angle. In addition to, the structure of the woven fabric, the applied surfactants improve the surface-particle binding within such fabric, which enhances the swiping stability and gives better results among the tested swipes. These studied characteristics seen to have improved the retention capability of the swipes when compared to the standard swipe TX304 tissue used by IAEA. Moreover, nesting certain complexing agents bound to the textile surface, tailor-made the selectivity of the swipe towards specific elements. This could be used to design purpose-fitting swipes. Standard TX304 swipe, can exhibit fairly high collection efficiencies for large particles from some surfaces, but for high

Z number, some of the adhered-dust containing particles can be lost during swipe handling or as a result of the bombarding electron beam during measurements.

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