

EVALUATING DESI IMAGING AND HIGH RESOLUTION MASS SPECTROMETRY TO IMPROVE AN INDUSTRIAL COMPONENT CLEANING PROCESS

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OVERVIEW

- DESI, Xevo® G2-XS QToF and HDI 1.4 were used to investigate an established industrial aqueous cleaning process.
- Components were directly analysed without extraction or sample preparation saving time.
- Combination provided information throughout the cleaning process, thereby facilitating process optimization and cost savings.
- Specific ions were localized to defined regions of the components that could be used to optimize their physical positioning in the cleaning process.
- This approach could be used to evaluate new cleaning processes with comparison to the existing process.
- Removal of surface contamination (soil, grease, oil, organic, inorganic material) is a critical step in the value chain in the manufacture of aerospace, automotive, medical device and semi-conductor products.

INTRODUCTION

The cleanliness of parts is critical to many advanced technology industries, impacting the viability of their products. Residual or introduced surface contaminants can dramatically alter the performance characteristics of critical components.^{1,2}

These industries generally use multi-step cleaning processes prior to assembly to assure the quality of their products. Process optimization often requires solvent extraction of the component after all or each of the cleaning steps followed by analysis of each extract.

Solvent extraction can be a time-consuming process that only reveals the identity and quantity of the bulk contaminants without revealing the location of those contaminants on the component. Solvent miscibility issues or drying regimes must be considered as part of the experimental design.

The combination of desorption electrospray ionization (DESI) imaging and high resolution mass spectrometry (HRMS) has been used to investigate a variety of surface materials.³⁻⁶ This combination offers a simple route to the visualization, identification and quantification of contaminants on the surface that may be used to optimize an industrial cleaning process.



Xevo G2-XS QToF fitted with a 2D DESI stage.

METHODS

Sample preparation

AISI316L stainless steel components, with a known contamination loading of a cutting fluid, Blasocut BC25MD, were cleaned using an aqueous based cleaning process.

Components were removed after each progressive step of the process and mounted together directly on the 2D DESI stage (Figures 1 and 2). DESI imaging with HRMS was performed without additional sample preparation.

DESI-MS conditions

MS system:	Xevo G2-XS QToF
Ionization:	2D DESI- (Prosolia)
Analyzer:	Sensitivity mode
Capillary voltage:	1.6 kV
Source temp.:	100 °C
Sampling cone:	79 V
Mass range:	m/z 50 to 2000
Scan rate:	3 spectra/s
Lock mass:	Leucine enkephalin (m/z 554.2615)
Gas pressure:	6.5 bar (nitrogen)
Solvent composition:	98:2 methanol:water (v/v)
Solvent flow rate:	1.5 $\mu\text{L}/\text{min}$
Spatial resolution:	300 x 300 μm
Stage speed:	900 $\mu\text{m}/\text{s}$

Data management

Data were acquired, processed, and reviewed using MassLynx[®] v4.1 and HDI v1.4 software. Regions of interest were defined on each component and then data compared using EZInfo (Umetrics) with principal component analysis (PCA).

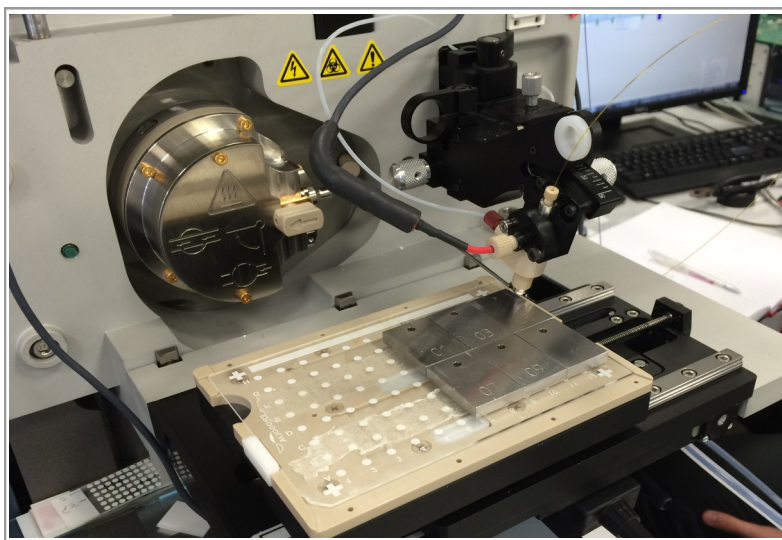


Figure 1. Stainless steel components sequentially removed from cleaning process and mounted on DESI stage.

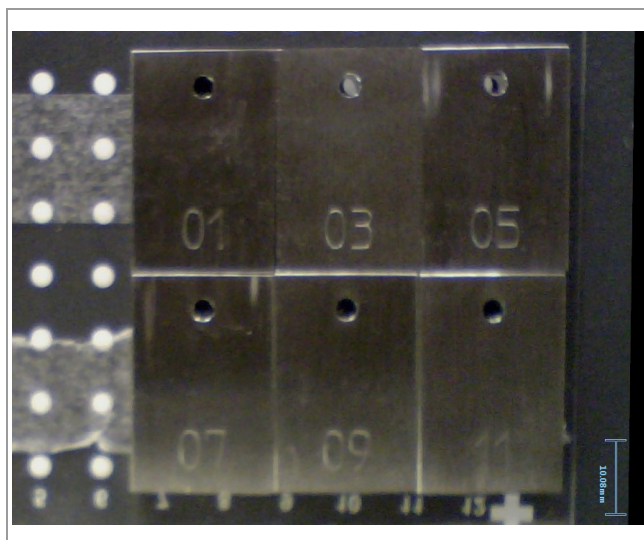


Figure 2. 01=Blasocut soaked, 03=1st rinse, 05=2nd rinse, 07=spray rinse, 09=3rd rinse and 11=4th rinse.

RESULTS AND DISCUSSION

Accurate mass ions were plotted according to their spatial distribution on the components. The 30 most abundant ions, ignoring lock mass, are illustrated in Figure 3.

Of particular note are the abundance of ions around the suspension orifice at the top of each component (especially 01, 03, and 11) and the fluid patterns on each component that have been lifted out of the rinse tanks compared to 07 which was spray rinsed.

Areas that displayed significant abundance differences were submitted to PCA. These included two areas each from components 01 (red area), 03 (light blue flow area), 07 (dark blue area) and 11 (light blue flow area). The scores plot of the pareto scaled PCA is displayed in Figure 4 showing good grouping between the two areas from each component yet good separation between each component.

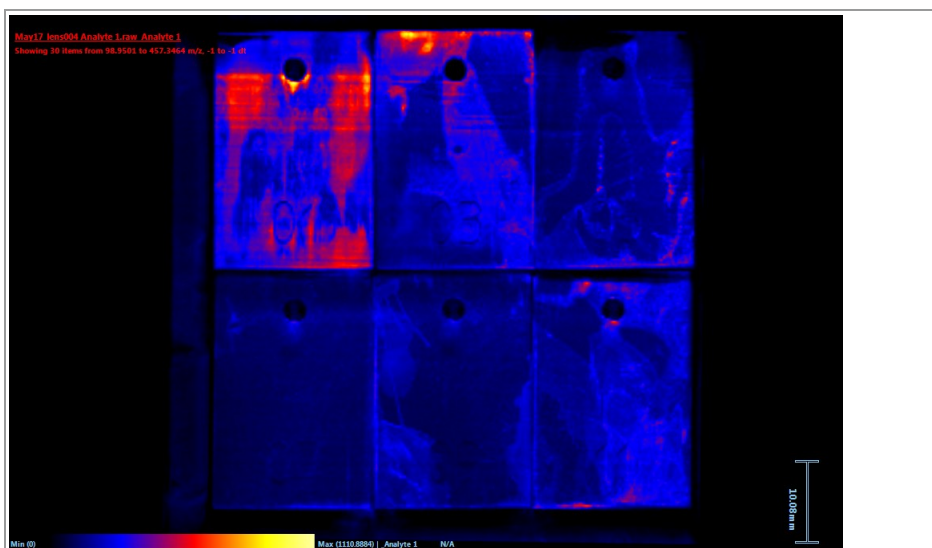


Figure 3. Image of 30 most abundant ions across six stainless steel components.

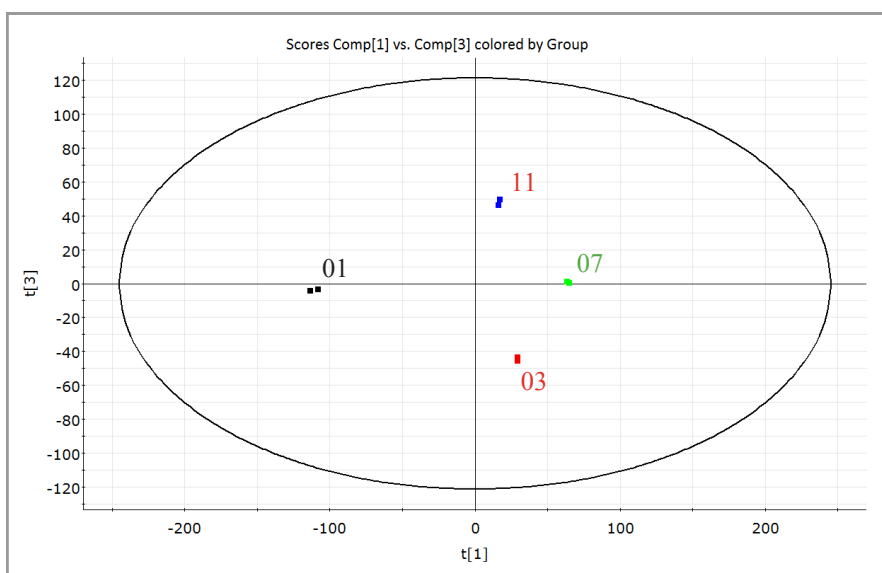


Figure 4. PCA of areas that displayed abundance differences between the components.

The images of the five most abundant ions responsible for the statistical differences between the areas selected on components 01, 03, 07 and 11 are illustrated in Figure 5.

As expected, the imaging dataset showed a significant decrease in ions associated with the cutting fluid, Blasocut, from component 01 that was proportional to the number of rinse and wash steps.

A few specific ions, particularly visible on component 07, remained on the components throughout the whole cleaning process. Some ions were introduced during the process which can be observed on component 11. If required, accurate mass of the ions can lead to elemental composition and subsequent identification through searching of online databases.

300 x 300 μm was chosen in this experiment balancing the speed of analysis and the level of local and spatial data required. All of this information would be lost if these components were solvent extracted.

Unwanted or unexpected ions may or may not lead to altered performance characteristics but knowledge of their existence and identity enable cleaning processes to be optimised to minimise their impact. Parameters such as ultrasonic frequency, immersion times, frequency of solvent and detergent changes, positioning and withdrawal are all variable.

This lack of knowledge and understanding of the cleaning steps often result in higher production costs and loss of product functionality.

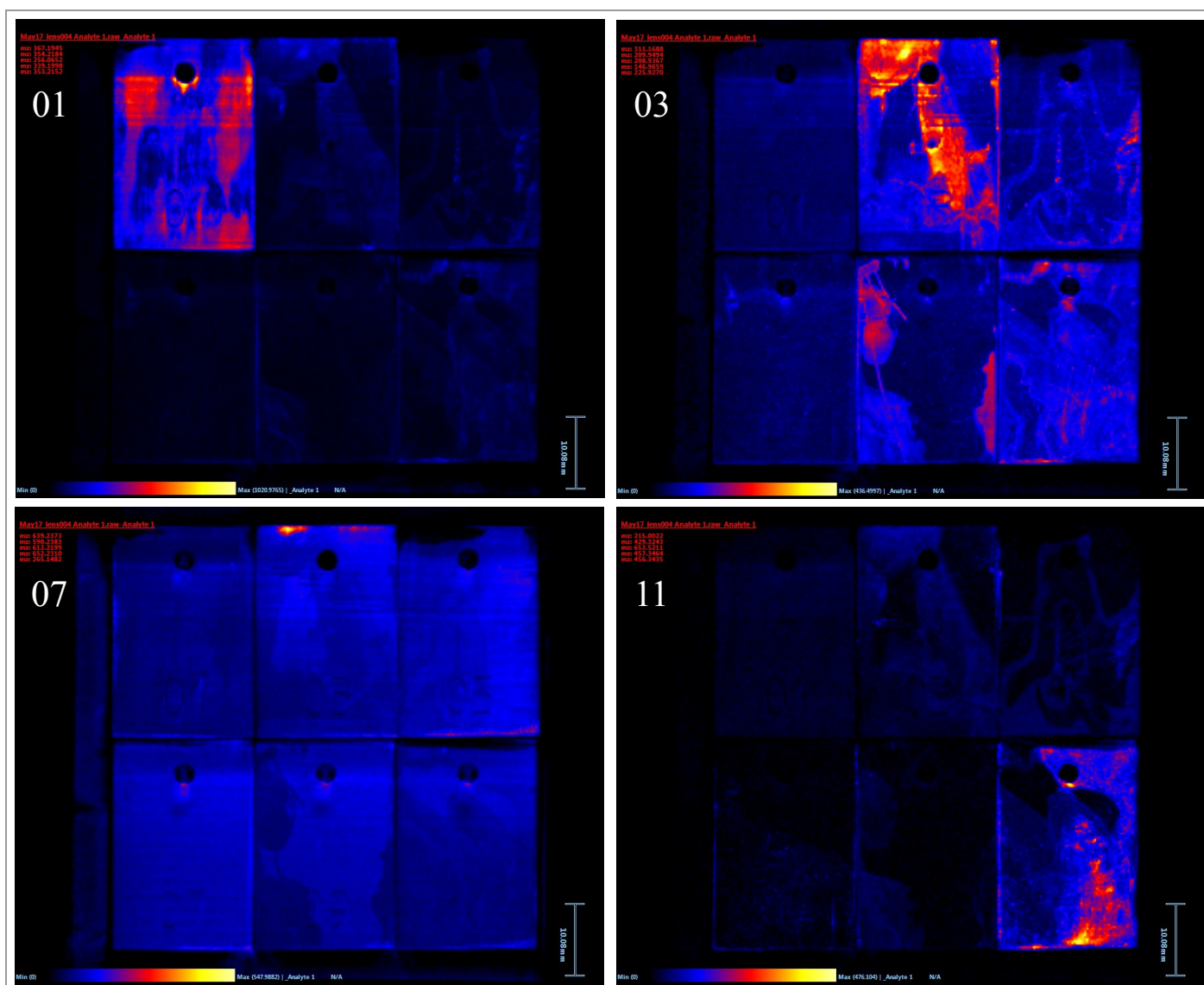


Figure 5. Image of 5 most abundant ions causing the statistical differences from each area of components 01, 03, 07 and 11 .

CONCLUSIONS

- An established industrial cleaning process was successfully investigated using DESI, Xevo G2-XS QTof, and HDI 1.4.
- Combination provided information about the process from the preliminary stages right through to final cleaning, thereby facilitating process optimization and cost savings.
- Components were directly analyzed without extraction or sample preparation leading to potential time savings.
- Specific ions were localized to defined regions of the components. This information could be used to optimize the physical positioning of components in the cleaning process.
- Results suggest that this approach could be used to evaluate new cleaning processes, as environmental guidelines or product performance requirements change, with comparison to the existing process.
- Removal of contamination (soil, grease, oil, organic, inorganic material) from surfaces is a critical step in the value chain in the manufacture of products in the aerospace, automotive, medical devices and semiconductor industries.

References

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