



UNIVERSITY OF  
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## PLASTER CASTS AND CORROSION

### AN EXPERIMENTAL STUDY ON REMOVAL OF CORROSION PRODUCTS FROM PLASTER CASTS



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Degree project for Bachelor of Science with a major in Conservation  
with Specialisation in Conservation of Cultural Heritage Objects

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## **ABSTRACT**

Conservation of plaster casts is complex due to the nature of the material itself. Corrosion of armature and metal structural supports is one of the most common causes of damage in plaster casts and subsequent corrosion staining. This study examines the use and efficiency of different cleaning systems in removing corrosion staining from plaster casts. Three different carriers were employed - commercial grade liquid latex, Arbocel® and xanthan gel - all in combination with varying concentrations of chelating agent EDTA. Evaluation of efficiency was based on ease of use, removal of corrosion products and damage to the plaster substrate. A combination of colorimetry, visual examination and documentation, microscopy and XRF analysis was used to evaluate results.

Results of this study demonstrate that corrosion staining in plaster casts can be treated efficiently with xanthan gels and Arbocel® poultices, albeit some original material may be removed in the process. The influence of prior cleaning and condition of the plaster object itself proved highly relevant. Latex cleaning systems proved unreliable and difficult to control on break edge surfaces; systems left residue and overall removed an excessive amount of original material. Arbocel® systems indicated the most potential; comparatively less damage to the plaster substrate and efficient removal of corrosion products.

It is further concluded that a combination of XRF and microscopy as a means of evaluating cleaning systems provided more supplementary data than colorimetry values - due to the surface stratigraphy of the experimental material and uneven severity of corrosion staining, reliable values were difficult to obtain.

Furthermore, results of this study strongly suggest that surface stratigraphy, substrate condition and extent of staining is highly relevant when choosing a carrier for delivering cleaning agents.

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## PREFACE

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# 1. Introduction

## 1.1 Background and context of the study

Plaster and plaster casting has a long history of use with evidence dating back to ancient Egypt. Casting of original sculptures to produce replicas was widespread during antiquity, with ample evidence from both Rome and Greece, and plaster was also used for model work and studies (Glombová, & Ďoubal, 2023a; Frederiksen & Marchand, 2010; Lee & Larsen, 1994). Little is known about casting during the middle ages; plaster work as ornamental decor from the 10th-13th centuries has been found in places such as Germany. During the 17th century, the use of plaster in interior stucco ornaments was widespread and quite common (Lee & Larsen, 1994).

The collecting and displaying of plaster casts was popularised in Europe during the Renaissance, many large collections originating in the 16th and 17th centuries. The spread and continued affluence of this trend is attributed to the timely reverence for classical art, and to the likes of King Louis XIV and Johann Joakim Winckelmann (Söderlind, 1999). During the 17th to 19th centuries, collections were amassed by nobles as well as institutions and casts of famous works of art could be found in art establishments as reference in anatomy studies (Frederiksen & Marchand, 2010; Payne, 2018; Söderlind, 1999). With the turn of the 20th century interest in plaster casts had started to fade. Plaster became seen as a cheap material, used in the process of making art, not intended for the art itself. Casts were deemed mere copies, and original artworks were prioritised (Söderlind, 1999). The decline of interest resulted in neglect and decay of many a large collections amassed during the *plaster cast boom* – if you will – and is a large contributor to the current state of plaster casts in collections, as well as the lack of research in conservation (van Beemen et al, 2013; Risdonne et al, 2022a).

Many plaster casts have internal structural supports made of a variety of materials such as wood, hemp and - of note for this study – armatures made of iron or steel (Risdonne et al, 2021). Corrosion related or corrosion caused damage in plaster sculpture and plaster casts are very common (Tišlová, Ďoubal & Zítková, 2023b). This is often due to poor storage conditions in damp environments with inappropriate climate and high relative humidity. Given the porous nature of plaster, when objects are exposed to high humidity this does not only affect the plaster itself; moisture permeates the material through capillary action and as a result the armature inside is also affected and may corrode (Tišlová, Ďoubal & Zítková, 2023a). Subsequent corrosion and expansion of metal armature causes a number of issues including breakage, cracking of and damage to the plaster mass, fragmentation, loss of mass, damages to and loss of surface treatment and/or paint layers as well as staining and discolouration of the substrate (Ďoubal, 2023c; Tislová, Ďoubal & Zítková, 2023b).

Fragments used in this study are part of a specific sculpture from 1843, made in the symbolic likeness of *Idun* – a character from Norse Mythology. More information regarding this work of art can be found under 2. *Background to the plaster sculpture Idun*.

## 1.2 Problem statement

Research regarding conservation of plaster casts is to date rather limited (van Beemen et al, 2013). When it comes to treatment and approach regarding corrosion related damage, few of today's existing studies address the issues that may arise with conclusive methods - this despite acknowledgement of damage caused as a result of corrosion being very common in plaster casts (Tišlová, Ďoubal & Zítková, 2023b). Decay and degradation of internal structural supports in plaster casts are among the primary causes of mechanical damage and metal elements such as armature – when corroding - are often the cause of the most severe issues (Tišlová, Ďoubal & Zítková, 2023b). In addition to cracking and loss of material, iron corrosion staining is a very common occurrence - and there are few conclusive studies on treatment methodology. The material plaster is in itself sensitive to moisture which makes it more difficult to clean, and increases the risk of corrosion in metal elements (Tišlová, Ďoubal & Zítková, 2023b).

Further research is required into both the practical and ethical aspects to lay the groundwork for future methodological guidelines for how to best treat issues such as corrosion staining in plaster casts.

## 1.3 Aim and research questions

Many materials and objects are highly sensitive to moisture and may react poorly when exposed to wetting and as such cannot be treated with aqueous methods without additional considerations (Chelazzi et al, 2018). The nature of plaster as a material puts casts in this category; the general consensus in current research is that wet methods are ill advised at best (Mrovč & Ďoubal, 2023; Risdonne et al, 2021). Gels and similar carriers can be used to deliver cleaning agents such as chelators, solvents and surfactants. These systems have a long history of use in conservation due to their properties such as limiting wettability and offering greater control over factors such as cleaning agent release, selective removal of soiling and local application (Bertasa et al, 2017; Chelazzi et al, 2018). Sequestering - or chelating - agents are frequently used in conservation to treat a number of issues, such as selectively removing materials with low solubility; dirt or old paint layers from paintings, corrosion products or other deterioration products involving metal ions (Rivers and Umney, 2003; Wilks, 1992; Wolbers, 2003). This study aims to evaluate the efficiency of three different carriers in combination with a chelating agent as treatment for discolouration caused by corrosion in plaster casts.

The following questions are what the study intends to answer:

- *Can corrosion products be removed with the selected methods?*
- *How effective are the different methods based on set parameters?*
- *Do the methods leave residue on the plaster substrate?*
- *Do either method damage the plaster substrate?*
- *Does the condition of the substrate influence the efficacy of selected methods?*

## 1.4 Scope and limitations

### ***The material***

The sculpture from which the material originates is in poor condition. It has suffered great damage, fragmentation, loss of material and is heavily soiled and dusty. No cleaning was done prior to testing; cleaning of plaster casts is in itself a complex issue and would have to be treated as a project of its own. Loose particles and dirt were removed with a soft brush where deemed necessary.

All material used for cleaning tests in this study are parts of an original historical object. The material consists of fragments taken from a sculpture with extensive discolouration due to corrosion staining and corroded armature. The material provided a realistic example; the extent to which corrosion products have migrated, as well as the time it would take would make it difficult to create realistically corrosion stained dummies within the allotted time frame. However this did also limit available surface area for cleaning tests and the experimental aspects of the study - such as time, exposure and areas tested - were adjusted accordingly. Results could also be influenced by the varying degree and extent of damage, presence of armature still stuck to the substrate and the unevenness of the surfaces overall.

### ***Analytical data***

Colorimetry is used to take note of changes in colour after treatment. Because of the origin and condition of the sculpture and thereby the fragment used it is not possible to determine, or consider what could have been seen as the original colours.

Regarding XRF analysis of cleaning systems: not all samples were analysed. The data included in this study is a selection thought to best represent the results.

### ***Plaster casts in history***

A brief summary of plaster casts and the collection thereof is featured in *1.1 Background and context of the study*. It is worth noting that the references used when summarising the history of plasterwork and plaster casting does not cover use outside of a primarily European context. Use outside of Europe has not been reviewed due to the context of this study and the origin of the fragments used.

## 1.5 Ethical framework

The following section offers a summary of key points in conservation ethics from literature and research regarding conservation principles and plaster cast conservation, as they relate to the current study.

Removal of corrosion products from plaster sculptures is generally done for aesthetic reasons as it often disrupts the intended display. As corrosion stains are generally considered a consequence of decay, they are not in accordance with an object's integrity as it is outlined in Applebaum (2007) and Muñoz-Viñas (2005). The *integrity* of an object is tied to its material properties, historical evidence, characteristic, and aesthetics (Muñoz-Viñas, 2005).

As outlined in Applebaum (2007) and Ďoubal (2023b), before any treatment is carried out an object's *integrity* needs to be considered - both material and immaterial aspects. Treatment should then be planned accordingly - balancing integrity, values and realistic conservation goals based on the context of the object and its values as a whole (Applebaum, 2007; Ďoubal, 2023b; Muñoz-Viñas, 2005). Recognizing that specifically for plaster cast conservation, due to the material complexity and the nature of many common types of issues, original material may be removed as part of any conservation treatment.

According to Phillips (1987) minimal intervention in regards to plaster conservation is an ideal - this could also be said about reversibility as any intervention involving cleaning does remove material (Muñoz-Viñas, 2005).

Corrosion stains are not part of the original intent nor *ideal state* (Applebaum, 2007) of plaster sculptures when they arise as a result of decay. Removing original material in this manner is done in order to preserve or restore part of the intended display, and as such must aim to balance object integrity through historical value, original intent and aesthetics.

Additionally, when considering treatment goals and what is achievable, the purpose of treatment and intended object use must also be taken into account (Ďoubal, 2023b).

## 1.6 Terminology and concepts

This section explains the use of some of the terms and concepts within the context of this essay. In addition, a handful of chemical terms are clarified as they may apply herein.

### ***Plaster***

In literature the term *plaster* and/or *plasterwork* can and does refer to both works made of gypsum plaster, lime plaster and other varieties (Lee & Larsen, 1994). Sculptures and casts are generally made from gypsum plaster, also known as plaster of paris. Within this essay, *plaster* refers specifically to plaster of paris, i.e. gypsum plaster.

### ***Plaster casts***

Casting and moulds have commonly been used both for the creation of original sculptures and for the making of copies and casts of original and historic works of art.

Within the context of this essay, *plaster casts* is used to refer to objects with the basis that they are most likely made by casting, whether it be original art or a cast thereof.

### ***Casting skin***

Refers to the surface layer of plaster formed when casting, closest to the mould. This layer is generally less porous and acts as a sort of barrier and/or protective layer. When damaged, additional issues are likely to occur. It is a direct translation of the Swedish term '*gjuthud*' (Pettersson, 2002).

### ***Corrosion related damage***

Here intended to encompass the wider variety of issues and damage that may occur as a result of corrosion in plaster sculpture. Generally speaking, in research and literature, the more commonly addressed issue is *staining*, that is to say surface discoloration due to corrosion products leaking or migrating. *Corrosion related damage* and/or *issues* is used within the context of this essay as a general term for any and all damages related to, or caused by corrosion. *Staining* is used to refer to discolouration caused by corrosion at surface level, and extensive staining caused by corrosion products having migrated further into the plaster substrate.

### ***'Original cast surface' and 'break edge'***

Refers to areas on the different fragments. Original cast surfaces are areas where the casting skin is mostly intact, and the surface intended for display. These areas are less porous, more even and some feature remnants of paint or previous treatments.

Break edges, as the term implies, are where fragments have broken from the plaster mass.

The surface is uneven, more porous than the cast surface, powdery to the touch and crumbles easily. Many of the fragments feature microcracks and fissures along the break edges, as well as small pieces of the original armature still lodged in the plaster at the break.

### ***Fragments B, S, and F***

Fragments used in the study are labelled based on their original positioning within the sculpture. B-fragments are part of the base, S-fragments part of the dress and/or skirt and F-fragments are part of the sculpture's feet. Due to the number of fragments labelled during the original assessment and documentation, the names and numbers have been kept as is in order to not be mixed up once returned to Samlingsenheten Stockholms Stadsmuseum. Photo documentation of the fragments, see *fig. 15-19*.

### ***Cleaning system***

Within the context of this study *cleaning system* as a term refers to the combination of solutions and carriers and additional material to form a tailored method intended to treat specific issues.

### ***Cleaning Samples***

*Cleaning samples* is used within this study to refer to the used cleaning systems post testing.

### ***Gel***

A gel is a *colloidal dispersion* of a solid component in an aqueous medium. Intermolecular interactions between components are what gives cause to gel-characteristics such high viscosity and low flow (Wolbers, 2017).

### ***Substrate***

Refers to the underlying surface of the treated fragments. Within the context of this essay *substrate* is used to refer to the plaster.

### ***Chelating agent***

A *chelating agent*, *chelator* or *chelate* are terms used to refer to a group of compounds that have the ability to form soluble complexes with metals in near insoluble compounds such as a variety of corrosion products. These agents are generally ligands containing a minimum of two functional groups that can function as electron donors (Rivers & Umney, 2003; Wolbers 2003). The term *chelate* stems from the Greek word '*chele*' meaning *claw* and is descriptive in the sense that chelating agents often bond around metal ions in a claw-like formation.

### ***w/v %***

Weight to volume percent. Used throughout this text to indicate ratio and concentration of a solid measured in grams mixed with an aqueous solution measured by mL.

### ***JP10oz/JP5oz***

Japanese tissue paper, or washi paper. It is made from kozo or gampi fibres and is commonly used in conservation for a wide range of purposes. *10oz* and *5oz* represent different grades of paper as expressed in standard *grams per square metre* (GSM), here in *ounces per yard*. In the following text *JP* will be used as an abbreviation for japanese tissue paper.



***Mohs hardness scale***

Mohs Hardness Scale is a scale system based on the hardness of certain minerals as standards. The scale goes from 1 – 10; Talc is the standard for 1, gypsum is the standard for 2, and diamond the standard for 10 (Rivers & Umney, 2003).

## 2. Background to the plaster sculpture *Idun*

The fragments used for the experimental aspects of this study all originate from *Idun*, a plaster cast sculpture currently in the care of Samlingsenheten Stockholms Stadsmuseum's collection. *Idun* and two sister sculptures were made in 1843 by Swedish sculptor and artist Carl Gustaf Qvarnström, according to information in Samlingsenheten Stockholm Stadsmuseum's object database. This information is further confirmed by Bengtsson (2003), adding that the sculptures were made near identical other than minor detail deviations. The sculptures were ordered by Fredrika Bremer, a famous Swedish writer known among other things for her novel *Hertha* and its influences on legal majority for women. *Idun* is a near life sized plaster cast, and was acquired by Samlingsenheten Stockholm Stadsmuseum in 1965 as part of a donation from *Fastighetskontoret, Bandhagen*. According to an *acquisition card* (fig. 1-2) located in the archives of Samlingsenheten Stockholm Stadsmuseum's library the sculpture had stood in their storage unit since the 1960's. Prior to this, *Idun* was located at *Villa Lyra* – a 19th century summer home, at the time managed by Carl and Frederika Linnell. The Villa was a known gathering place for many cultural figures around 1870-1880 (Stadsholmen, 2013). The card includes photo documentation which shows *Idun* in an unknown location, with some damage being visible even then; several fingers are broken on one hand, the other hand is missing, and there are clear indications of corrosion damage having already begun around the base (fig. 1-2).



Fig. 1-2 - Photos of *Idun's* acquisition card, found in Samlingsenheten Stockholm Stadsmuseum's library. Stockholm Stadsmuseum. SSM 31130.

*Idun* is in poor condition (fig. 3-4) and has suffered a variety of damage; most notably the sculpture is broken into a multitude of fragments. It is not clear when this occurred - as in the photographic evidence from the acquisition card *Idun* is mostly intact (fig. 2). The cast is partially hollow with armature made from iron, of which the majority was placed in the sculpture's legs and base. These structural supports are heavily corroded to the point of deformation and loss of material. Deformation has resulted in additional breaks of the plaster mass as well as severe discoloration and staining, particularly around break edges near where armature was originally present. All fragments of the sculpture are heavily soiled and there are deposits of ingrained dirt as well (fig. 4-7).



*Fig. 3 - Photo of Idun on location, in Samlingsenheten Stockholm Stadsmuseum's warehouse. The sculpture is broken in two larger pieces as well as a multitude of smaller fragments. Stockholm Stadsmuseum. SSM 31130.*



*Fig. 4 - Detailed photo of one of the larger pieces of Idun; featuring the sculpture's torso and head. One hand is missing and the other has several fingers broken off. The surface is heavily soiled. Stockholm Stadsmuseum. SSM 31130.*



*Fig. 5 - An assortment of fragments from Idun, showing the varying sizes and types of damage, including several pieces with visible corrosion stains. Stockholm Stadsmuseum. SSM 31130.*



*Fig. 6 - Deformed armature protruding from one of the sculpture's feet as well as parts of the fractured base. Stockholm Stadsmuseum. SSM 31130.*



*Fig. 7 - Deformed armature protruding from one of the sculpture's feet. Break edges are clearly visible where the plaster has fractured as a result of corrosion. Stockholm Stadsmuseum. SSM 31130.*

### 3. Previous research

The following chapter is intended to provide a summary of previous research reviewed within the scope of the study and said research's relevance to the subject. The summary is divided into sections and each section aims to address specific aspects of research as clarified in respective subheading.

#### 3.1 Conservation issues

Multiple studies within relevant research discuss the different conservation issues and aspects with plaster casts related to the material and its properties. The decline of interest in plaster casts is regarded in current research as a majorly contributing reason for the conditions of many collections today; a lack of priority resulting in poor storage, lack of conservation and proper preventive treatment as well as poor handling (van Beemen et al, 2013; Kłosowska & Obarzanowski, 2010; Risdonne et al, 2022a).

Risdonne et al (2022a) state that there is a certain lack of historical documentation surrounding techniques and treatments because of decline in interest; that this has contributed to the lack of expertise and limited research regarding conservation of plaster objects.

Kłosowska & Obarzanowski (2010) underline that in regards to the conservation issues, plaster casts are highly complex objects and reference the material's porosity as a major cause. As plaster is hygroscopic and very porous, the material attracts both dirt and moisture (van Beemen et al, 2013; Kłosowska & Obarzanowski, 2010). Similar arguments can be found in Tišlová, Ďoubal and Zítková (2023a), Risdonne (2022a) and Payne (2018).

Several studies also point out the sensitivity and 'low hardness' of plaster with regards to handling and mechanical influences, including the ease with which objects can be abraded during cleaning and other types of treatment (van Beemen et al 2013; Kłosowska & Obarzanowski, 2010; Mrovč & Ďoubal, 2023).

The combined lack of research and complexity of the material and its issues makes it all the more difficult to find appropriate and viable methods for conservation of plaster objects (van Beemen et al, 2013).

#### 3.2 Conservation treatments

As mentioned above research into methods for removing corrosion stains in plaster casts and sculptures is limited; among the methods mentioned in literature are chemical treatments involving carriers such as different gels and poultices (Mrovč & Ďoubal, 2023).

The methodology for related materials such as marble artefacts commonly involves chemical treatments of similar nature; Campanella et al (2022) investigate the removal of iron corrosion stains from marble with key focus on greener alternatives in regards to chelating agents. Their research offers insight into the use of both chelating agents and cellulose based poultices for treating iron corrosion stains in stone artefacts.

### *Cellulose-based poultices*

Cellulose-based products such as Arbocel® have high absorption capacities; there is some division on Arbocel® and water retention, approximately five times the weight according to Campanella et al (2022), and 4,5 according to Vergès-Belmin et al (2011).

There have been several studies published on the use of chelating agents and cellulose-based poultices as treatment for conservation issues such as staining from iron corrosion on marble sculptures and salt extraction in stone, and wall paintings (Campanella et al, 2022; Heritage & Bourgè, 2011; Spile et al, 2016; Vergès-Belmin, 2011).

Vergès-Belmin et al (2011) offer a summary on the use of different types of cellulose poultices in conservation and their applications. The study states there are controversies regarding the appropriate use of these methods, as well as listing reasons for how common their use; for example, properties such as neutral pH and ease of use. The research presented investigates the use and properties of Arbocel® like porosity, and the relevance of substrate pore distribution and pore size in regards to efficacy. Though the focus is primarily on the use of cellulose poultices for salt extraction, their research is relevant for the present study due to its detailing of Arbocel® and its properties as a commercial product.

Spile et al (2016) investigate the use of Laponite poultices and chelating agents for removing rust stains from marble. Their research offers insight into corrosion mechanisms and products that affect marble sculptures as well as treatments for rust staining on similar materials.

Kröner et al (2016) evaluate use of different poultices on desalination of stone based on poultice type, pore size distribution and method of application. One of the poultices examined is cellulose based, and the research paper features relevant information regarding poultice-substrate interactions.

The study includes arguments for the use of Japanese tissue paper as a means of separating poultice and substrate surface; if the condition of the substrate is such that methods could potentially cause a loss of material, Japanese tissue paper can act as a barrier and prevent damage during treatment. Other reasons for using tissue paper include pore size of substrate, surface stratigraphy that may cause cleaning materials to leave residue and substrate water sensitivity.

### *Gels and chelating agents*

The use, properties and applications of chelating agents in relation to plaster cast conservation are elaborated upon in several studies – relevant for the current essay were Doherty and Rivers (2017) and Wolbers and Little (2004). Both studies detail the cleaning of plaster casts through use of material analysis and cleaning systems involving chelates.

In Doherty and Rivers (2017) xanthan gel systems were used in combination with chelates and other solvents in order to remove layers of lead-based paint from a large plaster cast.

Their reason for using xanthan gel was largely due to its pH tolerance; the study emphasises the use of pH buffering to tailor the cleaning systems - ideally to effectively remove paint and dirt with minimal substrate interference. The study elaborates on the relevance of testing in order to adjust cleaning systems accordingly, as well as the importance of pH in relation to chelating agents, complex formation and solubility. Despite the primary focus being that of removing lead-based paint, their use of *Ethylenediaminetetraacetic acid* (EDTA) in combination with xanthan as well as their motivations and arguments for how the cleaning

systems were tailored and appropriate gel consistency provided relevant insight for the present study. It is worth noting that during the process, multiple applications of cleaning systems were performed and at a certain stage EDTA was excluded due to the risk of complex formation between chelate and calcium ions in the plaster substrate. Similarly, Wolbers and Little (2004) used chelating agents as part of cleaning systems when treating two painted plaster busts. Various analytical techniques were used to identify material components in the surface layers - dirt and paint - and in the plaster mass. Their research additionally provides relevant reasoning for the use of analytical methods, compatible with those presented in Risdonne et al (2021). Contrary to what is recommended in literature, Wolbers and Little relied on aqueous methods and swabbing. According to Mrovč and Ďoubal (2023) the use of cotton swabs is viable as a method of application when cleaning with aqueous solutions, although there is a notable risk of abrasion due to the softness of plaster as a material.

Although the research focuses on surface cleaning of plaster casts, it does provide contemporarily relevant insight on the use of chelates in plaster cast conservation, particularly in regards to considerations for complex formation, pH buffering and the complexities of plaster as a material. Additionally relevant for the present study are the discussions and arguments on tolerable concentrations of EDTA - without cleaning more than intended - and the use of pH buffering to optimise complex formation. Additionally, their research utilised spectrophotometry to evaluate efficiency of different cleaning systems through comparison of brightness values.

Mrovč and Ďoubal (2023) refer to a recent study on treating corrosion in plaster casts in which a mixture of chelating agent, Arbocel® BC1000 and Kelzan® HP-T was used with positive results. The latter ingredient consists of industrial grade xanthan gum, intended to act as thickener or stabiliser. In addition, Mrovč and Ďoubal (2023) note on studies done with citric acid - a chelating agent - applied as a poultice; reporting efficiency when attempting to treat corrosion staining on marble. It is here noted that for application on plaster, buffering would be necessary since too acidic a cleaning system could damage the substrate.

### *Latex films*

Regarding latex films as a method for treating corrosion staining in plaster casts, no previous research was found - however there is research concerning cleaning of casts and other stone surfaces using similar methods.

The use of film forming aqueous polymer dispersions, specifically for cleaning of plaster casts, are mentioned as an alternative in Mrovč and Ďoubal (2023). Using latex films to remove dirt and deposits as a method for conservation is additionally mentioned in Horie (2010). According to Mrovč and Ďoubal (2023), noted advantages to these systems are their film forming abilities, added controllability and potential use on localised areas.

Noted disadvantages are that the systems are difficult to control, and as additionally addressed in Nessow (2010), that they risk damaging the substrate by removing original material. Many latex-based products contain ammonia and as a result have a high pH, which may pose a risk to plaster surfaces (Mrovč & Ďoubal, 2023; Nessow, 2010).

There are several latex-based products available for the purpose of conservation, such as Arte Mundit® (Mrovč & Ďoubal, 2023; Nessow, 2010; Stancliffe et al, 2005) and Anjusil® (Risdonne et al, 2022b).

Arte Mundit® is a latex based product that was developed specifically for a large-scale project - the cleaning of the interior stonework in *St. Paul's Cathedral* – as detailed in Stancliffe et al (2005). The research includes properties, development and functionality. As the product is latex-based and the idea behind it is similar to the experiments carried out in the current study, it provided relevant insight into scientific and practical aspects of cleaning methodology when using latex-based systems. The experimental aspects and results in Nessow's bachelor's thesis from 2010 regarding the working properties of Arte Mundit® on wall paintings provided interesting perspectives on surface-poultice interactions.

Arte Mundit® is available in several versions that also contain EDTA. When applied to an object the aqueous part evaporates, leaving a thin polymer film that adheres to dirt and particulate matter on the surface of the substrate. This film can subsequently be peeled off taking adhered particles with it (Nessow, 2010; Stancliffe et al, 2005).

Risdonne et al (2022b) make use of Anjusil® in cleaning plaster – it is used specifically to remove localised deposits of dirt on 19th century plaster casts of decorative details from Notre Dame.

### 3.3 Analytical methods

Risdonne et al (2021) account for the application of different analytical methods used to identify the chemical components of a 19th-century plaster cast; among the methods used are *Fourier Transform InfraRed Spectroscopy* (FTIR) and *X-Ray Diffraction* (XRD). The study offers concise explanations of what has been analysed, for what purpose, as well as the arguments and reasons behind the choice of method. For example regarding the use of FTIR; analysis indicated the presence of organic materials in the surface layer. Prior research have given several examples of organic mediums used with different purposes in plaster casts (van Beemen et al, 2013; Glombová & Ďoubal, 2023b; Tišlová, Ďoubal & Zitková, 2023a).

Identification of materials in plaster casts aids in understanding the extent of damage, causes of decay, and can be utilised as a basis when planning treatment. The use of lead based paint was not uncommon in plaster sculptures, an example of which is described in Doherty and Rivers (2017) and other surface treatments occur in many forms, for example linseed oil, gum arabic and beeswax. The presence of either could influence the result of cleaning methods; lead for example, can form complexes with chelating agents, and as such could interfere with the desired complex formation.

Risdonne et al (2021) state that their study is in part intended to showcase just how material analysis of plaster objects may be used and for what purpose; parallels can thusly be drawn to Doherty & Rivers (2017), Wolbers & Little (2004) and Bruni et al (2022).



## 4. Theoretical background

The following chapter covers the scientific background of aspects relevant to the study. This includes the material properties of plaster and plaster casts as well as how they relate to corrosion staining, properties and working mechanisms of chelating agents, and the carriers used herein. For the present essay it is of importance to better comprehend the various degradation mechanisms and how these relate to material properties, as well as how different components influence the condition of casts over time. The various material components in plaster casts as well as their composition and purpose will influence the processes of decay and degradation - specifically in regards to the relationship between material properties of plaster casts and corroding armature.

### 4.1 Material properties of plaster casts

Plaster of paris, or gypsum plaster is made through burning of the naturally occurring mineral *gypsum* (Risdonne et al, 2021). Burn temperature, the rate of heating, mineral origin, impurities and additives all influence the properties of the plaster in finished casts (Tišlová, Ďoubal & Zítková, 2023a).

Gypsum consists primarily of *calcium sulphate dihydrate*,  $\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$  – that is to say water is bonded within the crystalline matrix, so called *crystallisation water* (Payne, 2019; Risdonne et al, 2021). When burned at a temperature of approx 105-160°C the compound loses water, and becomes *calcium sulphate hemihydrate*,  $\text{CaSO}_4 \cdot \text{H}_2\text{O}$ . To use in casting, this is mixed with water to the point of slight oversaturation, cast and left to set. The hemihydrate is subsequently reformed and during setting water evaporates - leaving behind a porous, solid structure. The reaction is exothermic, that is to say it produces heat, and is occasionally referred to as the plaster 'burning' (Pettersen, 2002). This is relevant in relation to moulds and deterioration; when plaster mass is poured into a mould the pressure build up caused by an ever so slight volume expansion causes the formation of what will henceforth be referred to as *casting skin*. This volume increase is approx 0,5 – 1 % v/v (Tišlová, Ďoubal & Zítková, 2023a). *Casting skin* forms as a layer where plaster mass meets mould; plaster crystals pack tighter and this results in less porosity (Tišlová, Ďoubal & Zítková, 2023a). This *skin* acts as a protective barrier of sorts for the more porous mass underneath. If damaged, this allows dirt and moisture to penetrate the underlying cast to a greater extent. When evaluating the condition of objects made of plaster it is of significance to consider the *casting skin* and whether or not it is intact (Pettersen, 2002).

Porosity is also related to how the gypsum was burned and at what temperature, and the amount of water added to the plaster mass prior to casting (Payne, 2019).

## 4.2 Corrosion in plaster casts

The following section describes the mechanisms of iron corrosion and the reactions that occur, and provides a summary of the type of damages that may occur in plaster casts as a result of corrosion.

### 4.2.1 Iron corrosion

Metal corrosion can be explained as a chemical reaction with the surrounding environment that leads to deterioration of the original material and its properties, as well as chemical changes (Rivers & Umney, 2003; Shreir, 2013).

Rust and corrosion are often used synonymously with oxidation and this is correct only in part. Corrosion reactions occur in so called *electrochemical cells* and are redox reactions - that is to say both reduction and oxidation are part of the process. For corrosion to take place there are certain general requirements as outlined in Selwyn (2004); an electrolyte that allows for ionic transport, a metal conductor that allows electron transfer, an anodic site and a cathodic site. The electrolyte is in many cases water; moisture buildup, localised condensation or rainwater, and not much is required. The anode provides a site for oxidation reactions; in the case of iron components, this reaction likely occurs between iron atoms and oxygen dissolved in water.

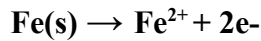
The rate and extent of corrosion as well as the resulting products are highly dependent on certain factors, such as humidity, pH, oxygen and other ions present such as air pollution or dirt containing chloride ions or sulphates (Campanella et al, 2022; Selwyn, 2004; Tišlová, Ďoubal & Zítková, 2023b). With iron, the primary reaction can be seen as  $Fe(s)$  oxidising - by giving up 2 electrons and forming  $Fe^{2+}$ . These ions can remain in solution and migrate into the substrate, or they may react further and form insoluble compounds and precipitate. Electrons may flow to the cathodic site where the reduction half-reaction takes place; these reactions vary depending on the metal, other ions and species present and factors such as pH of the surrounding environment and electrolyte itself.  $Fe(II)$  can be further oxidised to  $Fe(III)$  if there is enough oxygen present in solution (Selwyn, 2004).

The reddish brown and orange corrosion products that are commonly associated with rust stains on plaster generally consist of various forms of iron hydroxides, oxides and hydrated oxides (Tišlová, Ďoubal & Zítková, 2023b). Spile et al (2016) offer the following reaction as a summary for the formation of *rust*.  $FeOOH$  is used here as a standin for various potential corrosion products of iron.



The initial oxidation half reaction can be found below. Electrochemical cell and moisture transport illustrated in *fig. 8*.

### ANODIC HALF REACTION



### CATHODIC HALF REACTION

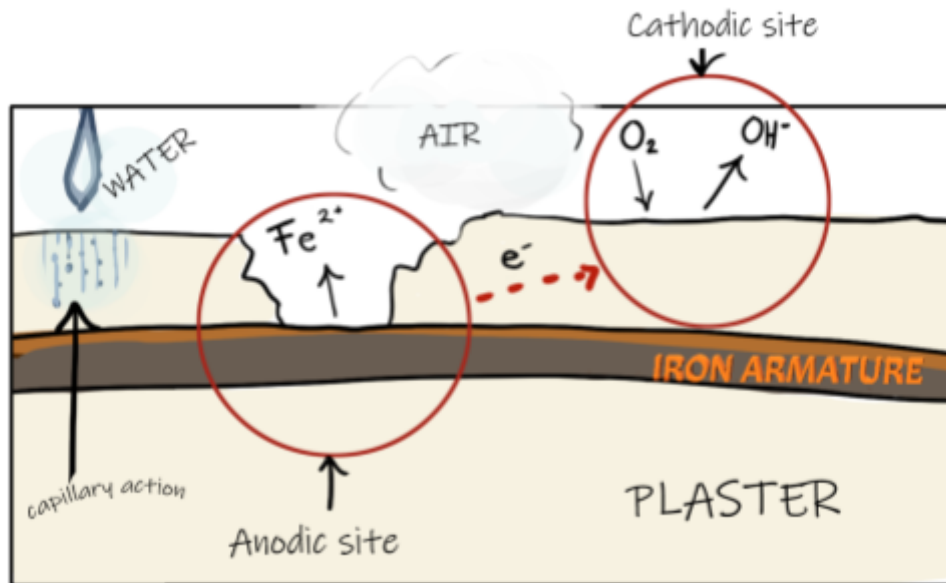
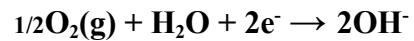


Fig. 8 - Illustration of an electrochemical cell and moisture transport in plaster casts.

#### 4.2.2 Corrosion related damages

Corrosion products of iron and steel are generally larger in volume than the original metal and as such require more space (Rivers & Umney, 2003). Plaster as a material is very soft, only 1.5 -2 on Mohs hardness scale; materials with the value of 1 on the scale can be scratched by a mere fingernail (Rivers & Umney, 2003; Tišlová, Ďoubal, & Zítková, 2023a). Due to plaster being a softer material, the expansion of armature can cause the plaster substrate to crack and fragments may break off entirely. If metal structural components are close to the surface this also increases the risk of damage when the metal corrodes. Corrosion may also result in loss of mass as well as damage to the cast surface, such as cracks to surface treatments, loss of casting skin and flaking paint, and corrosion products can easily migrate further into the substrate causing additional discolouration (Ďoubal, 2023c; Tišlová, Ďoubal & Zítková, 2023b).

## 4.3 Chelating agents

### 4.3.1 Chelating agents

The term chelating agents, or chelates, refers to groups of organic acids. They are so called *ligands* due to having unbound electrons in their molecular structure that allow the formation of soluble complexes with ionic species such as otherwise insoluble metal salts (Rivers & Umney, 2003; Spile et al, 2016; Wolbers, 2003).

In conservation, chelating agents are often used as part of tailored cleaning systems intended to treat specific issues; removal of soil and corrosion products are common examples (Rivers & Umney, 2003; Stoner & Rushfield, 2012). There are many different kinds of chelators and their properties and affinities for complex formation with different metal ions vary greatly; examples of commonly used chelates are EDTA (*Ethylenediaminetetraacetic acid*), HEDTA (*Hydroxyethylethylenediaminetriacetic acid*) and citric acid. The chelates themselves are water soluble, and when ionised in solution can attract metal ions through opposing charges (Stoner & Rushfield, 2012). Chelates can be categorised as bi-, tri- or polydentate structures in accordance with the amount of functional groups available to coordinate and/or bond to a metal ion (Wolbers, 2003). As stated above (*see 1.6 Terminology and concepts*) chelating agents in essence work as claws or cages – they can form complexes with metal ions through functional groups acting as electron donors. The complex formation is considered an equilibrium reaction and as such changes with the environment; conditions such as pH influence the outcome (Rivers & Umney, 2003; Wolbers, 2003).

In simple terms, due to molecular composition and the presence of polar molecules, acids ionise in solution. The degree of ionisation depends on the strength of individual acids, as well as pH of the solution itself. The chelating effect of EDTA and similar compounds equally depend on pH, due to ionisation. The more ionised the acid, the more available bonding sites (Wilks et al, 1992). Complex formation affinity and binding force are dependent on the amount of available binding sites with the chelate. As chelation is influenced by pH, buffering to values appropriate for complex formation and substrate interaction is often required (Rivers & Umney, 2003). These properties in chelating agents are what make them useful to conservators - cleaning systems can be tailored to interact with specific species and thereby limit damage to the substrate. When choosing a chelate one must generally consider both the nature of the substrate as well as the type of soil or deterioration one intends to remove.

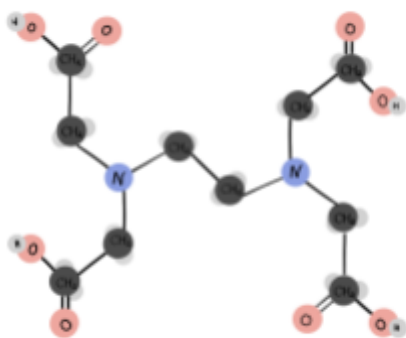
### 4.3.2 Ethylenediaminetetraacetic acid (EDTA)

*Ethylenediaminetetraacetic acid*, *EDTA*, is a chelating agent with the chemical formula  $C_{10}H_{16}N_2O_8$ . It is a weak acid, and strong chelator due to its molecular composition (*fig. 9*). EDTA is commonly used in conservation as well as other fields such as dentistry, textile production and in medicine (Mohammadi et al, 2003; Waters et al, 2001; Man et al, 2018).

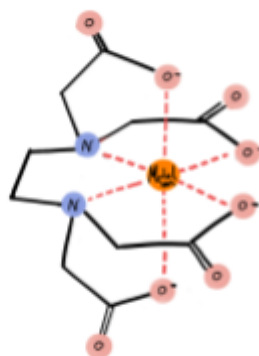
Research in fields outside of conservation is mentioned here due to relevance when selecting chelating agents for the present study. In conservation, EDTA is commonly used to remove corrosion products on different materials such as iron artefacts and sculptures made of marble and similar materials (Cuvillier et al, 2022; Spile et al, 2016; Wilks et al, 1992). Other uses are removal of paint layers, as exemplified in Wolbers and Little (2004) and Doherty and Rivers (2017).

EDTA offers six potential sites for ionic bonding (*fig. 10*), and due to its properties as an acid the ability to form complexes is dependent on degree of ionisation. pH is therefore a relevant factor (Wilks, 1992; Wolbers, 2003).

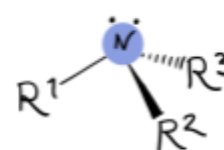
An EDTA molecule has four carboxyl groups and two tertiary amines (*fig. 11*). In aqueous solution EDTA ionises in stages, depending on pH. EDTA requires a pH above at least 2 before any kind of chelating effect can take place (Rivers & Umney, 2003). At pH 6,2, the acid is fully ionised and chelation is at its peak (Wolbers & Little, 2004).



*Fig. 9 - Illustration. Molecular structure of EDTA.*



*Fig. 10 - Illustration. Claw-like behaviour of an EDTA-molecule in an EDTA-metal ion complex.*



*Fig. 11 - Illustration. Tertiary amine.*

#### 4.4 Carriers

The use of cleaning systems like gels and poultices give far better controllability than simply using cleaning agents in solution; evaporation rate of solvent into substrate can be minimised, treatment time adjusted. Wetting of substrates that may be more sensitive to moisture can also be limited and better controlled using gels or poultices. These systems also facilitate cleaning of sensitive materials with varying degrees of damage, as they allow for localised treatment to a greater extent than aqueous solutions alone (Bertasa et al, 2017; Rivers & Umney, 2003). Aqueous systems on water-sensitive surfaces can be risky; due to the sensitive nature of plaster and its porosity, aqueous methods are ill advised; gels and similar systems are a good alternative (Mrovč & Ďoubal, 2023).

#### 4.4.1 Arbocel®

Arbocel® consists of crystalline cellulose fibres, ground into powder and is manufactured and sold by Deffner & Johann (Vergès-Belmin et al, 2011). The product is available in multiple versions – differing in fibre length. In this study Arbocel® BC200 was used; it has an average fibre length of 300 µm, and average fibre thickness 20 µm (TDS).

Properties of cellulose poultices cited by previous research as beneficial include neutral pH, minimal residue on objects after treatment, high absorption and ease of use (Campanella et al, 2022; Vergès-Belmin et al 2011). Poultice formation of cellulose powders is due to absorption of moisture into the fibres.

#### 4.4.2 Xanthan

Xanthan is a polysaccharide of high molecular weight with the monomer chemical formula  $C_{35}H_{49}O_{29}$ . The compound is soluble even in cold water, and can form gels at a wide range of pH values due to its structural configuration (Giraud et al, 2021) (fig. 12). It is a polymer consisting of repeating glucose units (Wolbers, 2017) also referred to in literature as a *pentasaccharide repeat unit* (Sutherland, 1993).

Xanthan gum receives its name from the bacteria it originates from; *Xanthomonas Campestris*. The bacteria produces xanthan in the form of secretions and the process of extraction involves several steps (Wolbers, 2017); these will not be elaborated upon here due to relevance. Commercial xanthan gum is sold in the form of a powder, and is used in industries such as food and cosmetics. It is organic, considered non-toxic and fit for consumption as an additive in food (García-Ochoa, 2000).

The pH tolerance of xanthan gels make them somewhat unique, and an appropriate choice when using chelating agents since chelating effects are pH-dependent. This allows for pH adjustment and thereby tailoring of cleaning systems based on chelators used, substrate composition and targeted ions for complex formation (Doherty & Rivers, 2017).

Type of bacterial strain and growth conditions cause variations in the molecular structure (Sutherland, 1993; Wolbers, 2017). Due to its molecular compositions xanthan gum has two possible configurations, depending on pH; a random coil configuration if pH is above 6, and an ordered helix configuration if pH is below 4,6. It is also possible for both configurations to be present at the same time. Because of these properties, gels can form at a wide range of pH (Angelova et al, 2017; Giraud et al, 2021).

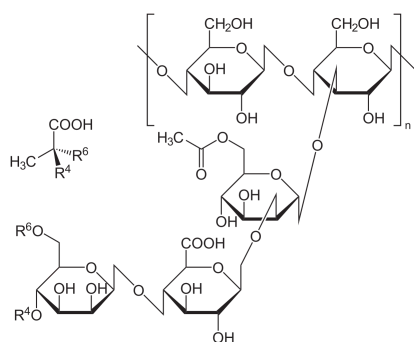


Fig. 12 - Structure of xanthan gum. Wikimedia Commons.  
<https://commons.wikimedia.org/wiki/File:Xanthan.svg>

### 4.4.3 Liquid latex

Commercial moulding latex generally consists of an aqueous dispersion of prevulcanized rubber (Horie, 2010). Vulcanization of rubber is in essence the process of heating rubber together with sulphur in order to cause cross linking. This is done to chemically strengthen the latex (Coran, 2013; Samsuri & Abdullahi, 2017). The latex itself originates from the bark of *Hevea brasiliensis*, and is an elastomeric hydrocarbon polymer consisting primarily of polyisoprene (Joseph, 2013). It is prevulcanized and contains approximately 60 % ammonia, which is added to prevent deterioration (Horie, 2010). The alkalinity of the resulting product is likely due to added ammonia.

## 4.5 Analytical methods

The following section offers brief explanations of the analytical methods used in this study; *Fourier Transform InfraRed Spectroscopy* (FTIR), *X-Ray Fluorescence* (XRF) and colorimetry measurements. The analytical methods were chosen based on their application in previous research on plaster casts, and colorimetry was used due to its application for measuring colour differences and providing evidential data, as noted Catenazzi (2017), Cuvillier et al (2022) and Wolbers and Little (2008).

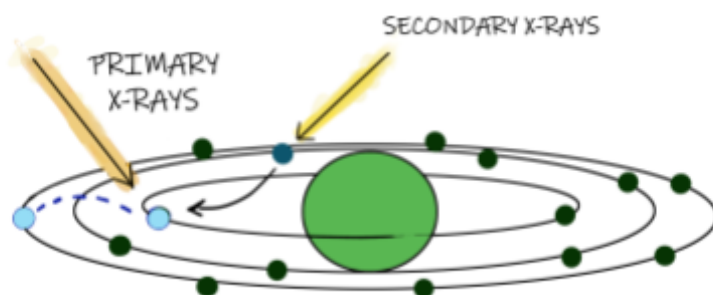
### 4.5.1 Fourier Transform InfraRed Spectroscopy (FTIR)

Information on the properties and working mechanisms of Fourier Transform Infrared Spectroscopy (FTIR) is explained in great detail in Derrick et al (1999), *Infrared Spectroscopy in Conservation Science*. The publication also features in-depth descriptions of how to interpret spectrums. FTIR is an analytical tool that utilises Infrared Light to identify the unique energy levels, and signatures of elements, and translates these into spectrums via software and mathematical processing. It is used for quantitative analysis of samples with organic components, and relies on the unique energy signatures of elements. As with XRF, FTIR also relies on electromagnetic radiation to produce spectrums like the ones obtained through material analysis – although the energy beamed at samples does not excite electrons, since it is not high enough. Intermolecular bonds give atoms in molecular configurations a certain degree of freedom and movement – it is the energy transitions of these movements that correspond with IR-radiation during analysis (Derrick et al, 1999).

#### 4.5.2 X-Ray Fluorescence (XRF)

XRF is a non-destructive analytical technique that can aid in establishing the components and chemical composition of inorganic materials in a wide range of samples (Shackley, 2011; Stuart, 2007).

In simplified terms, an X-ray tube is used to beam samples with a ray of high energy, the primary; causing electrons in the sample atoms to jump from inner energy levels to outer ones (*fig. 13*). As atoms strive towards stable energy states, this will cause quite the opposite by creating vacancies in the inner levels – this will cause electrons in the outer shells to move down to fill them; emitting energy in the form of corresponding X-rays (Hitachi High-Tech, n.d.; Stuart, 2007). These X-rays are produced due to the difference in bonding energy between outer and inner energy levels, and they are of lower energy than those initially beamed at the sample (Shackley, 2011). The energy emitted is unique for each element; and is picked up by a detector in the equipment. These secondary X-rays are translated into spectrums via computer software. Rays emitted are commonly classified as K, L and M, and additionally as alpha and beta depending on which shell they correspond to – and are labelled as such in spectrums (Stuart, 2007). XRF can provide both quantitative and qualitative results - elements present and their amounts in the analysed sample (Shackley, 2011).



*Fig. 13 - Illustration. Simplified workings of primary and secondary x-rays.*

#### *Margin of error*

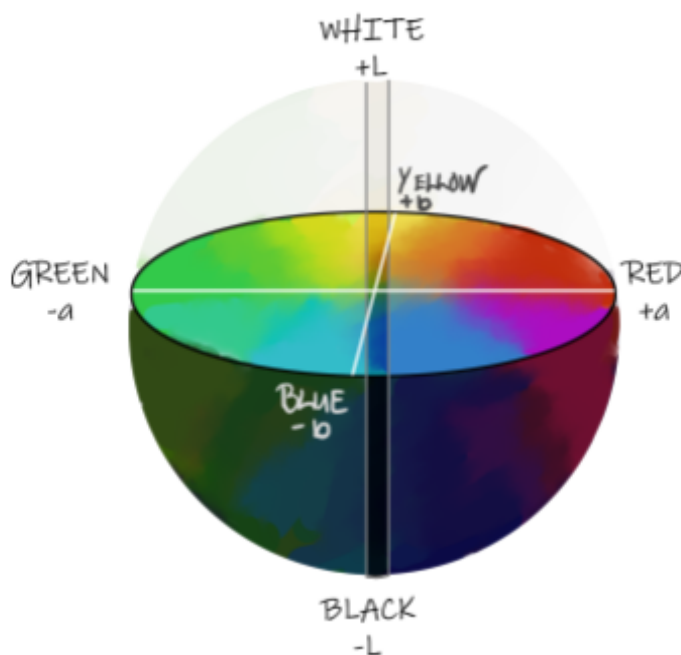
Due to results, it is necessary to consider margins of error for XRF; results of XRF analysis and the limits of material identification are dependent both on equipment, and on sample composition. One such error that may occur, and could be applicable here is *spectral line overlap*. Line overlap may occur due to elemental components in a sample, for instance if their energy positions are too similar (Hitachi High-Tech, n.d.; Gallhofer & Lottermoser, 2018, Thomsen et al, 2006). In regards to equipment, factors that may influence this include characteristics and settings such as optics and resolution (Thomsen et al, 2006). Shackley (2001) further elaborates on so-called *matrix effects*; overlap and mass absorption effects that can occur due to variable mineral compositions. Mass absorption results in altered intensity, and can occur due to coexisting elements and their influence on radiation absorbance.



There are methods for stripping overlap, however - analyses done in this study were done on a tight schedule with equipment designated to be used by multiple students. As such, no stripping routines were employed.

#### 4.5.3 Colorimetry

Colorimetry is a way of measuring colour spaces. The CIELab system from 1976 gives coordinates that represent different values in the colour space (*fig. 14*).  $L$  represents lightness,  $a$  and  $b$  represent colour on the scale of greenness-redness and yellowness-blueness respectively (Johnston-Feller, 2001; Saunders, 2020). When using colorimetry to compare colours, the difference can be calculated through these values for  $\Delta E$ .  $\Delta E$  is an expression of the total colour difference (Johnston-Feller, 2001). There is some discrepancy in literature as to the perceivable deviations for  $\Delta E$ ; on average, deviations above 2 could be considered visible upon close inspection (Catenazzi, 2017).  $L$  values range from white (100) to black (0), positive values for  $\Delta L$  - differences in  $L$  values from one sample to another - indicate an increase in brightness (Cuvillier et al, 2022; Saunders, 2020).



*Fig. 14. - Illustration. Colour space and associated values.*

## 5. The Study

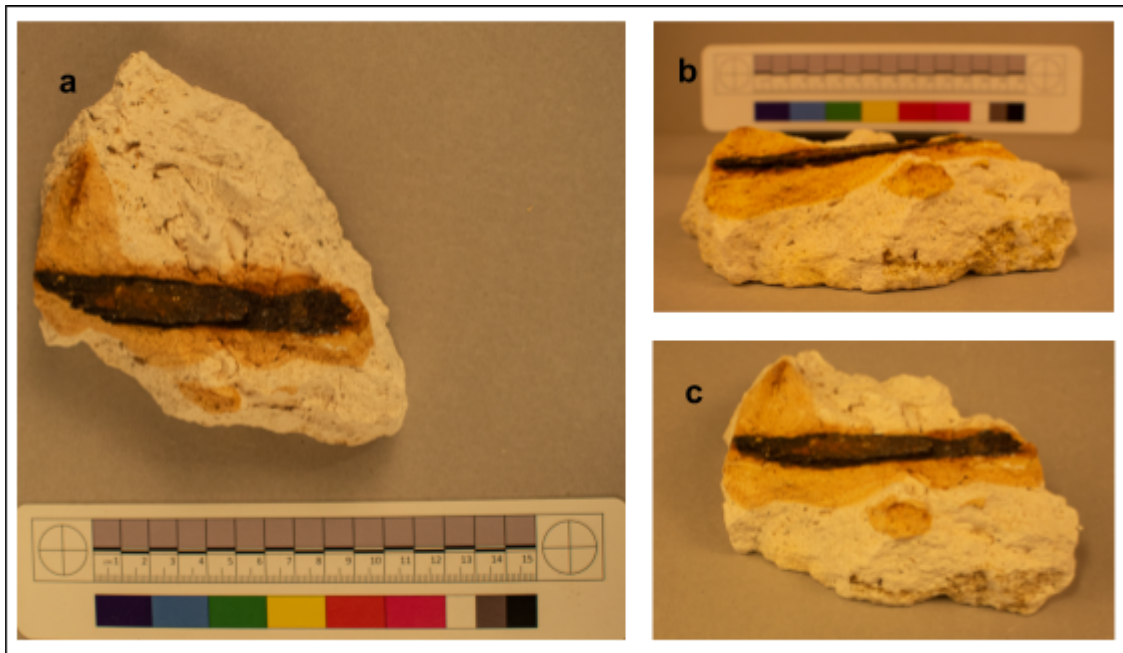
### 5.1 Materials and method

#### 5.1.1 Fragment selection

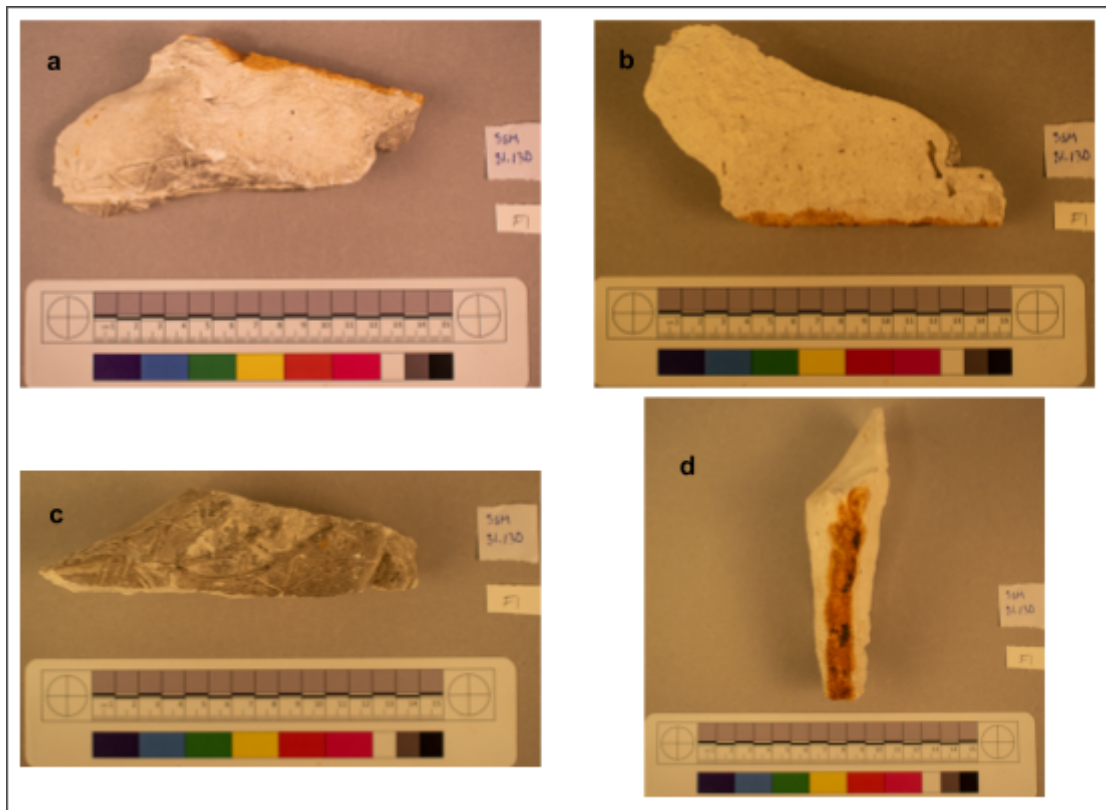
A total of 5 fragments from the sculpture *Idun* were selected for cleaning tests. The fragments all have varying degrees of staining and migration of corrosion products into the plaster substrate, as well as varying extents of damage. The fragments vary additionally in that some feature parts of the original cast surface and some feature primarily fractured surfaces and break edges.

Fragments *B3* and *B6* are parts of the sculpture's base. Fragments *F1* and *F5* are part of one of the sculpture's feet. Fragments *S11* and *S9* are parts of the sculpture's robe.

- Fragment *B3* (*fig. 15*) has a larger piece of corroded armature still stuck in the substrate. Staining is located on the fractured surface of the break edge, and in the area closest to the lodged piece of armature.
- Fragment *F1* (*fig. 16*) has several smaller pieces of armature along the break edge in addition to staining. The top side on *F1* is part of the original cast surface; the *casting skin* is intact, with some minor staining as well as flaking paint and dirt present.
- *Fragment B6* (*fig. 17*) has several smaller pieces of armature lodged in the substrate along the break edge, as well as staining in the same area. The fragment is taken from inside the sculpture's base, and features a clear example of migration of corrosion products along the sides of the fractured edge.
- *Fragments S11* and *S9* (*fig. 18-19*) do not feature corrosion related damages – they were used to test cleaning systems for adherence and ease of removal prior to testing on the studied corrosion staining. These samples were also used in material analysis.



*Fig. 15a-c - Fragment B3, prior to cleaning tests.*



*Fig. 16a-d - Fragment F1, prior to cleaning tests.*

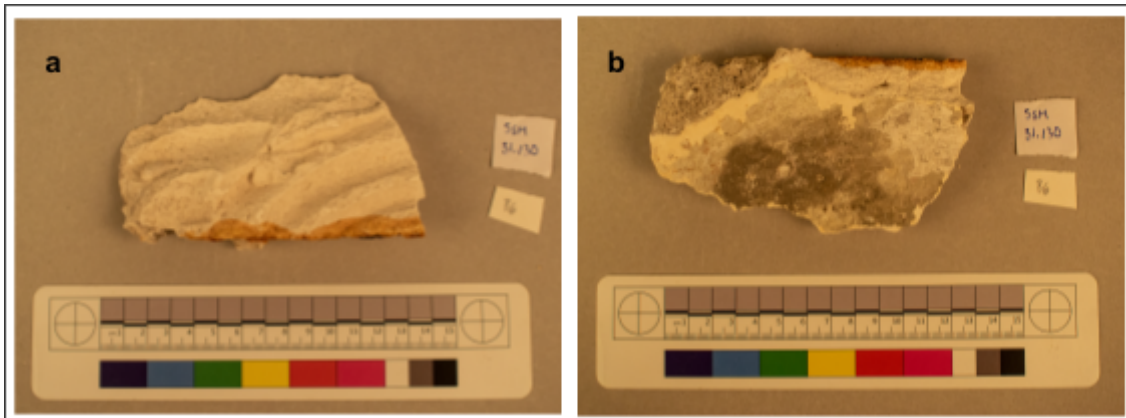


Fig. 17a-b - Fragment B6, prior to cleaning tests

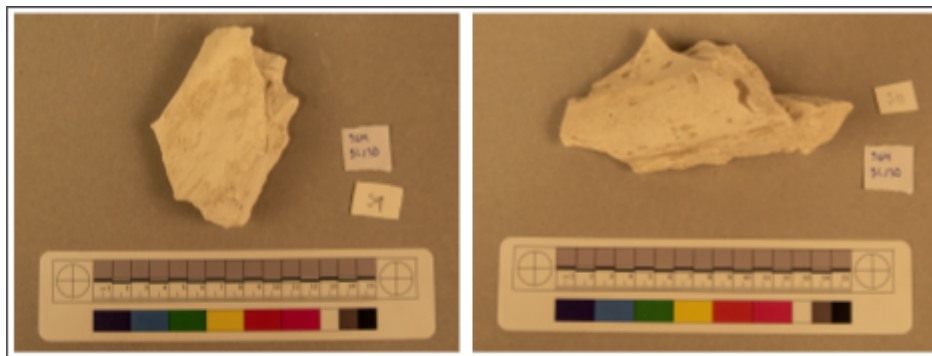


Fig. 18 - Fragment S9, used in material analysis and during preliminary testing.

Fig. 19 - Fragment S9, used in material analysis and during preliminary testing.

## 5.2 Analysis and documentation

### 5.2.1 FTIR

FTIR-analysis was done in order to attempt to identify potential surface treatments used during casting as well as the components of the lower paint layers. Material was taken from fragments *S11* and *S9* (fig. 18-19). For equipment, see *Appendix I*. Little could be said about potential surface treatment - results were inconclusive in that regard. FTIR analysis of substrate proved useful in other aspects - results are a close match to database spectra for gypsum plaster. This became relevant when comparing results of XRF analysis, see *6.5 XRF analysis*. For the FTIR spectrum, see *Appendix V*.

### 5.2.2 XRF

Analysis was done on a sample of armature (*fig. 20*) to confirm it was made from iron. Additional analysis was done on *S11* and *S9* to determine the composition of the plaster substrate and if other inorganic components were present such as additives used to alter properties during casting.

A selection of cleaning samples were also analysed with XRF to identify what had been removed. Samples were selected based on what would likely give the clearest results, i.e. those of the different carriers with the highest evaluated removal of either substrate or corrosion products, compared with other samples of the same cleaning system.



*Fig: 20 - Armature fragment used in XRF analysis.*

### 5.2.3 Colorimetry

A *Konica Minolta CM-26d Spectrophotometer*, together with the *Colibri*® software, was used to measure colorimetric values from several areas on *B3*, *F1* and *B6* before and after cleaning tests..

Each area, as illustrated and numbered in *fig 62*, was measured thrice and average values calculated. Colour differences for each areas  $\Delta E$  were calculated according to the 1976 CEIL  $a^*b^*$  equation formula expressed below (Johnston-Feller, 2001).

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^{*2})]^{1/2}$$

A list of all values can be found in *Appendix II - Colorimetry Data*. For specifics on equipment, see *Appendix I*.

## 5.3 Method

### 5.3.1 Selection of cleaning systems

Though there are many types of chelates with differing properties, EDTA has a long history of use and is a recurring example in conservation studies as well as in other fields of research (*see 4.3 Chelating agents*). It is considered a strong chelating agent and the majority of sources reviewed for this study surrounding the topic of chelates feature examples and information regarding the use of EDTA. In addition, it is the chelating agent most familiar to the author of the current study, and one that was readily available within the allocated resources and facilities. Due to the limited time and scope of the study it was deemed the most appropriate choice. A solid base in previous research regarding properties and behaviour also provides a greater variety of reliable sources when interpreting results. Carriers were selected based on previous research and available materials, as well as the desire to evaluate systems with different properties.

Xanthan gum was selected due to its ability to form gels at a wide range of pH values as well as its use in previous studies. Cleaning systems that can tolerate different pH values are relevant for future research into plaster cast conservation as they allow for buffering and adjustments in accordance with different chelators and substrate composition.

Gel viscosity can also be adjusted with relative ease; due to the nature of plaster substrates and different corrosion products it may be of interest to future treatments if the pH of gel systems may be adjusted to suit both, and viscosity can be adapted to better suit surface and break edge stratigraphy.

As presented above (*see 4.4.1 Arbocel®*), Arbocel® and the application of cellulose-based poultices as carriers in cleaning systems has a good basis in previous research regarding similar materials and issues. Arbocel® differs from xanthan and latex in both properties and preparation and additionally, falls in the category of poultices once prepared rather than gels - and as such provided an interesting alternative.

Latex was selected due to similar systems such as Arte Mundit® and their presence in relevant research. This would also offer an interesting alternative to xanthan gel and Arbocel® as it functions neither as a gel nor poultice, but rather forms a thin film on the surface of the substrate. Evaluating how the latex interacted with the stratigraphy of a fractured surface was additionally relevant as gel systems such as agar and xanthan have been known to leave residue (Giraud et al, 2021; Mrovč & Ďoubal, 2023).

JP was used in all cleaning tests due to the water sensitive nature of the plaster substrate and because the break edge surfaces were uneven, with a multitude of micro-cracks and pieces of armature present - all elements that could cause cleaning systems to get stuck and leave residues. The break edge surfaces were fragile and the use of JP as a barrier between substrate and cleaning systems minimised the risk of loss of original material, as noted in Kröner et al (2016).

The following systems were used in this study:

- Xanthan gel 7% (w/v) with 5% EDTA (aq), pH 6,2
- Xanthan gel 7 % (w/v) with 2% EDTA (aq), pH approx 7,8
- Xanthan gel 3,5 % (w/v) with 5% EDTA (aq), pH 6,2
- Xanthan gel 3,5 % (w/v) with 2% EDTA (aq), pH approx 7,8
- Liquid latex with 5% EDTA (aq), 1:1
- Liquid latex with 2% EDTA (aq) 1:1
- Arbocel® 200BC with 2 % EDTA (aq)
- Arbocel® 200BC with 5% EDTA (aq)

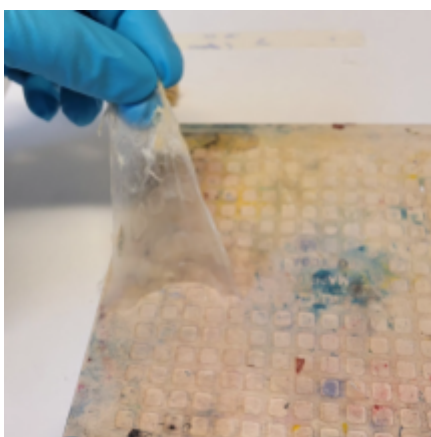
The combinations of carriers, chelators and pH values were limited due to resources and time available. Commercial grade liquid latex intended for arts and crafts was used with the sole intention of evaluating its efficacy together with EDTA on break edge surfaces.

All pH measurements were taken using pH strips; they provided reliable results and using more elaborate equipment for precise values was not possible with the allocated time and the resources available.

### 5.3.2 Preliminary testing

Prior to cleaning tests on the selected fragments, pre-testing of both latex and xanthan systems were performed. These included mixing of gels for appropriate viscosity as well as determining whether or not the latex could be used as a carrier and still be viable for intended purposes. With the test mixtures of liquid latex and EDTA solutions - if not stirred immediately, a reaction between the components resulted in a semi solid precipitate clumping to the stirring tool.

Application and removal were tested first on a soiled tile, due to its porous and uneven nature (*fig. 21*), followed by tests on fragments from *Idun* with similar surface stratigraphy to that of the studied fragments (*fig. 18-19*). Latex was applied to the test surfaces with a soft brush and left to rest for 20 min. A flexible plastic spatula was used to loosen the edges of the film, and it was carefully peeled off the substrate. Initial testing gave positive results with little to no residues (*fig. 21*).



*Fig. 21 - Peeling of latex film from test surface.*

Preliminary testing of xanthan gel (*fig. 22*) without JP showed difficulty in removal without leaving a substantial amount of residue and minor but significant damage to the substrate. Using JP as suggested by literature (Kröner et al, 2016) allowed for more even and controlled application of gel and latex systems, as well as easier removal. The tissue paper was thin enough to allow the latex mixture to flow smoothly and follow the stratigraphy of the surface well (*fig. 23*).

JP of two different degrees, 10oz and 5oz, were selected due to influence on substrate-system interactions with the cleaning systems tested.

Tables for the application of each cleaning system with times of exposure, fragments tested and number of applications can be found in *Appendix II - Cleaning tests*.



*Fig. 22 - Preliminary tests for xanthan and Arbocel® systems on fragment S11.*



*Fig. 23 - Preliminary tests of latex systems on fragment S9.*

### 5.3.3 Buffered EDTA solutions

Two solutions of EDTA were prepared to 2 % w/v and 5% w/v respectively, at a total volume of 200mL each, by dissolving EDTA salt in deionized water. During preparation the solutions were buffered with 2 M NaOH in aqueous solution to set pH values. 2 % EDTA was buffered to approx pH 7,8 & 5 % EDTA buffered to pH 6,2. EDTA is most effective past its third dissociation constant, approx pH 6 (Wolbers, 2003).

The buffering of the second solution to 7,8 was done to evaluate the effect of slightly more alkaline conditions.

Further adjustment of pH values could have been done to tailor the solutions more specifically to increase the possibility of chelation with iron, and reduce the risk of complexes forming with calcium. However, that is not within the scope of this study. The study is designed specifically to determine whether or not the tested methods are viable and therefore does not explore tailoring the methods further and does not take factors such as *conditional stability constants* into consideration.

It is equally worth noting that buffering was done solely with xanthan gel in mind due to its affinity for forming gels under a wide range of pH values, and as such latex and Arbocel® were not considered and their original pH values were not taken into account. Arbocel® has a pH between 5-7 according to manufacturer (SDS) - which also matches values measured



during preparation. The addition of buffered EDTA solutions did not indicate any significant changes to pH. The liquid latex has a rather high pH; likely due to ammonia content. Measurements indicated pH 9,5-10 and the addition of buffered EDTA solution did not indicate any changes.

#### 5.3.4 Cleaning systems

##### *Xanthan gels*

Two primary batches of xanthan gel were prepared according to instructions found in *The John and Mable Ringling Museum of Art: Modular cleaning recipes* (2016), to a concentration of ca 7 % w/v; 3,74 g of xanthan gum powder with EDTA solution to a total volume of 50mL respectively. Thorough stirring by hand was required in order to achieve a usable gel; the reaction between xanthan gum powder and aqueous solution happens quickly, and the mixture clumps easily. Results of the first cleaning tests with xanthan showed the viscosity of the gels was too high for it to work effectively through the JP barrier.

For further testing of xanthan gel, secondary batches were prepared by diluting with additional EDTA solution to 3,5 % w/v xanthan with 5 % and 2 % EDTA respectively. The secondary batches proved more efficient; lower viscosity allowed the gel to flow with more ease and thus follow the stratigraphy of the surface better. These versions of the gels were also able to work more efficiently through the JP barrier.

##### *Liquid latex*

Liquid latex was mixed directly with 2% EDTA and 5 % EDTA solutions respectively. Both combinations were mixed to a ratio of approximately 1:1 - 20mL liquid latex to 20mL EDTA solution. The mixtures were thoroughly stirred by hand using a glass stirring rod. Ratios are approximate because of difficulty in measuring the liquid latex due to texture and flow, affinity for foaming, as well as the presence of precipitate if not mixed fast enough. The precipitate would form as a single rubbery mass, approximately 1-2 cm across. It could easily be removed with a pair of tongs. Since the latex starts to set albeit slowly, once mixed in an open vial, several batches had to be made over time. Precipitation did not occur in all batches. The amount of precipitate differed between batches when it did occur, and prior to application to fragments said precipitate was always removed. This would ultimately alter concentration and ratio. The presence of precipitate did not seem influenced by the EDTA concentration nor pH of the added solution.

##### *Arbocel®*

Arbocel® poultices were prepared in accordance with standard ratios (Campanella et al, 2022; Vergès-Belmin et al, 2011); 1 g Arbocel® 200BC powder to 4-5 g EDTA solution, 2% and 5% respectively. In total approximately 18-20 grams of each combination was prepared and left to rest for between 2-12 hours.

## 5.4 Cleaning tests

All cleaning tests outside of preliminary testing were done using JP, either 10oz or 5oz, as a barrier between substrate and cleaning system. The JP was first divided into smaller sheets; size appropriate for the intended test areas with additional margins of approximately 0,5 cm on all sides. Sheets were then positioned on the intended fragment and area individually prior to application of cleaning systems for each test.

All used cleaning samples were marked and kept separately for examination and documentation to follow. From here on out the different cleaning systems and used samples thereof will be referred to by their combinations of carrier, grade of JP used and EDTA concentration. For combinations used on each fragment, times of exposure and number of applications see tables in *Appendix II - Cleaning tests*.

### *Xanthan*

Primary mixtures of 7 % xanthan gel, both with 2% and 5% EDTA were applied to select areas on *B3*. 3,8% xanthan gel, with 2% EDTA and 5% EDTA respectively were applied to select areas on *B3*, as well as select areas on *B6* with additional tests performed due to altered viscosity. Application was done by first positioning JP on intended areas, then applying the gel with a soft flat head brush and plastic spatula. Time intervals for xanthan were set to 1hr after preliminary testing; less than 40 minutes did not give the system enough time to have any measurable effect, and too long a time risked the gel setting past the point of easy removal. Gel was removed with the aid of a painter's knife and thin plastic spatula, by carefully lifting the edge of the JP and easing both gel and paper off the surface of the substrate.

### *Latex*

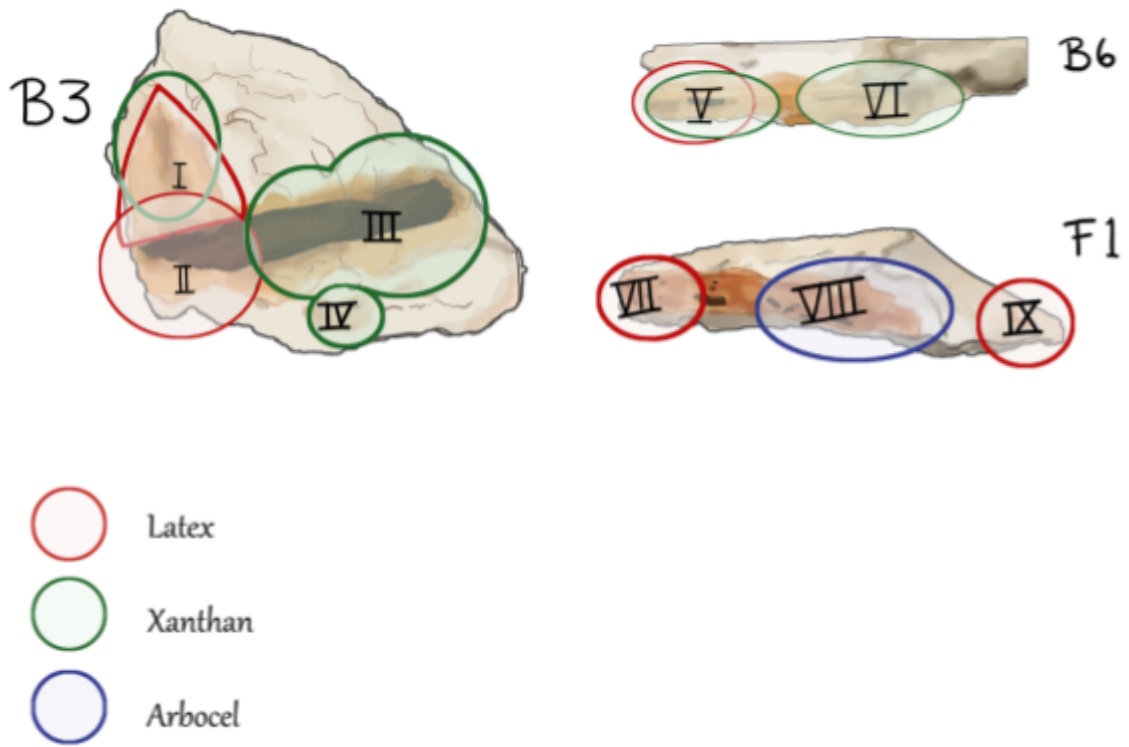
As with xanthan gels, JP of appropriate size was first placed to the intended area, followed by using a soft flat head brush to apply the latex system on top.

Time intervals were evaluated and adapted throughout testing due to how the systems behaved and interacted with the substrates varying surface stratigraphy. Latex systems were applied to select areas on *B3*, *B6* and *F1* - including staining on the original cast surface on *F1* (*fig. 16*). Time intervals were set to 3-5 min, 10-20 min, 30 min and 1 h.

### *Arbocel®*

*Arbocel®* systems followed the same procedure in regards to JP. The poultice was carefully smeared onto the paper with a slim flatheaded plastic spatula. Removal was performed by carefully taking hold of the paper edges, lifting both poultice and paper off the substrate. Time intervals were evaluated and altered during experiments based on substrate-poultice interaction, poultice drying times and substrate stratigraphy; 15-20 min, 45 min and 1h. All combinations of *Arbocel®* systems were tested on *F1*.

Test areas for the different systems on each fragment are shown below, in *fig. 24*.



*Fig. 24 - Illustration showing where on each fragment the different systems were tested.*

## 6. Results

Results of each cleaning system and combinations with EDTA and JP are presented below. The parameters used to evaluate efficiency for each system are as follows: corrosion product removal, substrate fragment removal and ease of use. In order to evaluate the efficiency in regards to the different parameters, several methods were utilised.

- Colorimetric values were used as a means for evaluating removal of corrosion products through changes in colour, comparing values taken before and after cleaning tests.  $\Delta E$  values provide data for the total colour change, and could aid in determining efficiency of the different systems.
- Microscopy was performed to examine the surface of each fragment before and after cleaning tests in order to evaluate the effect each system had on the substrate surface in regards to removal of plaster fragments and potential damage to the surface of the substrate. In addition, microscopy provided visual evidence for determining whether or not the different systems left residue on any of the fragments.
- Microscopy, visual examination, and documentation of all cleaning samples were performed in order to evaluate the extent of corrosion product and substrate particles removed, and to better compare the results of the different combinations and systems with each other.
- XRF analysis was performed on select samples to identify the chemical composition of the material removed from the fragments, i.e. to provide data representing percentage plaster and iron present on the cleaning samples. Results of analysis provided data that could be used in combination with visual examination and microscopy of samples in order to establish efficiency of the different systems, as the data received could be used as further evidence concerning mass of plaster and corrosion removed from the surface of the plaster substrate.

### *Time of exposure*

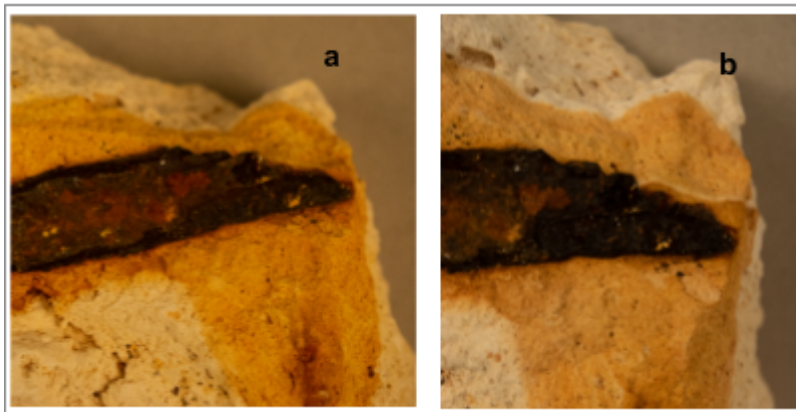
Initial testing indicated xanthan gels needing approximately 1 hr to yield significant results; reducing time of exposure as done with latex and Arbocel® methods was deemed irrelevant. Latex sets relatively quickly, with visible results on corrosion products after a short amount of time - 3-5 min - though testing showed differences in both corrosion and substrate interactions with longer exposure. Arbocel® similarly starts to visibly dry after approximately 15 minutes; though as shown below longer time of exposure yielded better results.

### *Original cast surface*

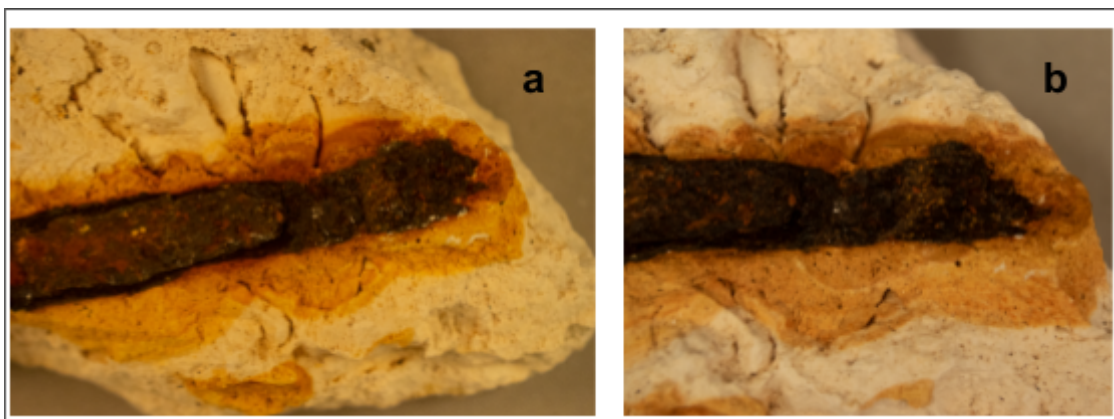
Only one of the fragments featured corrosion staining on the original cast surface (*fig. 16*). Due to the difference in substrate surface and results from break edge cleaning tests, latex systems were used on this area. Arbocel® systems were also used on this area due to the minimal substrate removal shown during testing.

## 6.1 Photographic documentation

Overall, when visually comparing results through before and after pictures of tested areas on all fragments, the difference in colour is slight, which does little to indicate efficiency in regards to corrosion stain removal. There is however a somewhat notable difference in surface grime and dust particles, albeit difficult to differentiate what is due to corrosion being removed and what is layers of dirt (*fig. 25-26*). Microscopy of tested areas also point towards removal of some grime and particulate matter, and as with photography and visual examination the surface effect on corrosion staining was not conclusive (*fig. 27-28*).



*Fig. 25 - Fragment B3, latex area, before (a) and after (b) cleaning tests. After pictures show a slight reduction in surface grime as well as minor colour differences.*

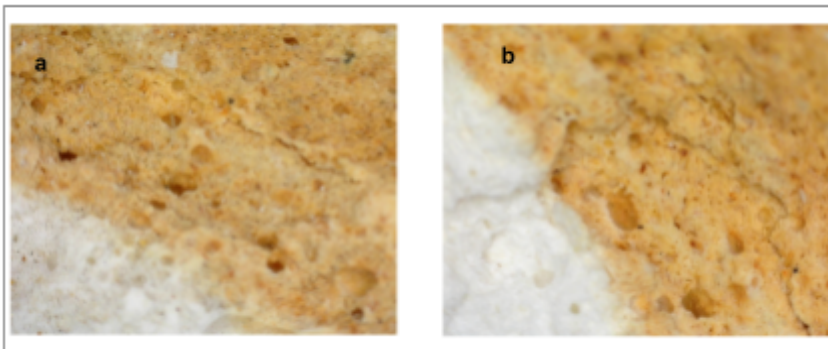


*Fig. 26 - Fragment B3, latex area, before (a) and after cleaning tests (b). After pictures show a slight reduction in surface grime as well as minor colour differences.*

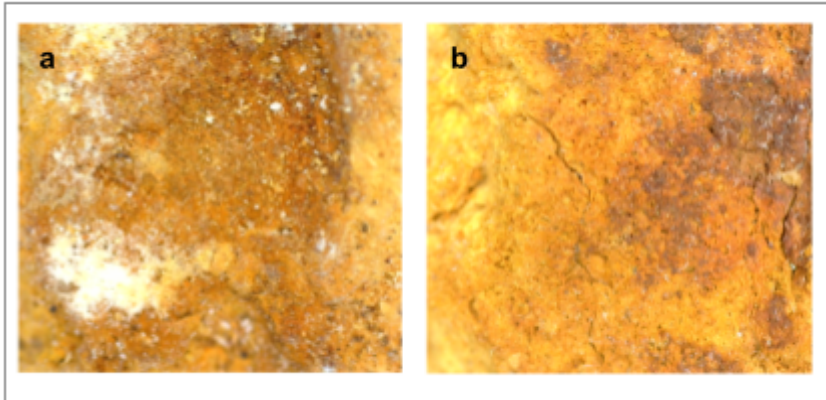
Minor residues from latex and xanthan systems were noted on all fragments - primarily from latex in areas where the cleaning system stuck to the plaster surface, as well as fibres - most likely from JP.

As for areas with pieces of armature still lodged along the break edges, there is clear evidence that some of these were removed - which could be either due to chelation effect or due to the adherence of cleaning systems pulling pieces out when removed (*fig. 29*).

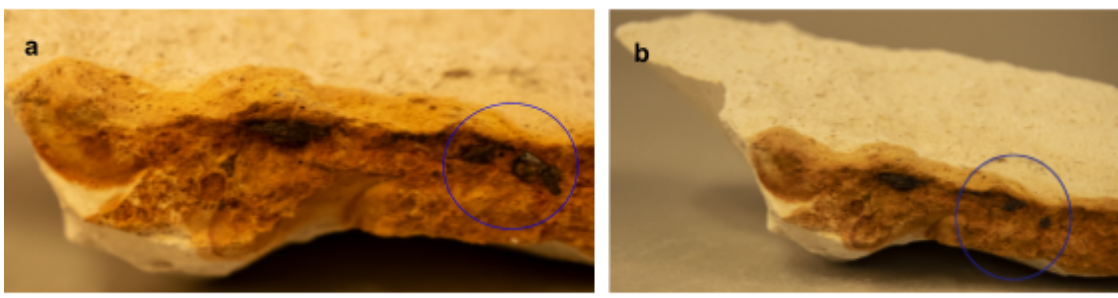
Picture documentation from cleaning tests with latex and Arbocel® systems on the original cast surface do show a clear reduction of corrosion staining in addition to removal of surface grime (*fig. 30*).



*Fig. 27 -  
Microscopy images of  
fragment B6, before (a)  
and after (b) cleaning  
tests.  
Magnification: 70x*



*Fig. 28 -  
Microscopy images of  
fragment B3, before (a)  
and after (b) cleaning  
tests.  
Magnification: 70x*



*Fig. 29 - Fragment F1 before (a) and after (b) cleaning tests. Blue circles indicate areas where armature pieces have been removed.*

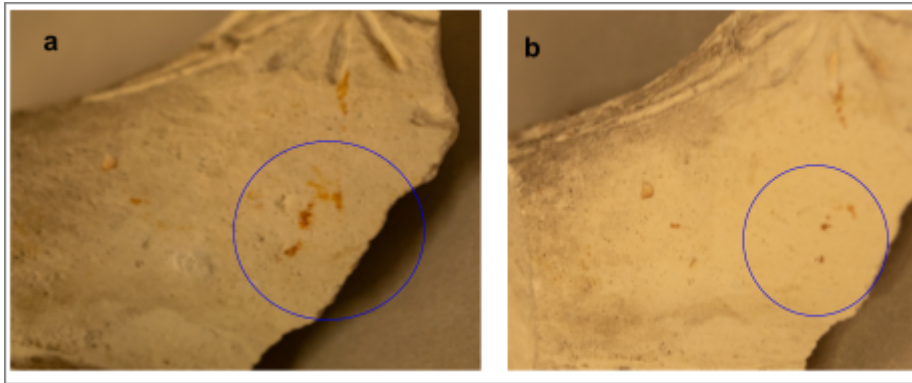


Fig. 30 - Corrosion staining on original cast surface, fragment F1, before (a) and after (b) cleaning tests.

## 6.1 Xanthan

Visual examination and microscopy imaging of xanthan samples indicate removal of corrosion products for all versions of the gel systems (fig. 32-37). JP as a barrier was necessary to avoid wetting and excessive residue; trial attempts without JP resulted in gels nearly impossible to remove. JP 5 oz broke easily, especially with the weight of the gel which increased the risk of residue as well as paper fibres sticking to the plaster. 10oz remained intact during application and removal, making the gels easier to use (fig. 31).

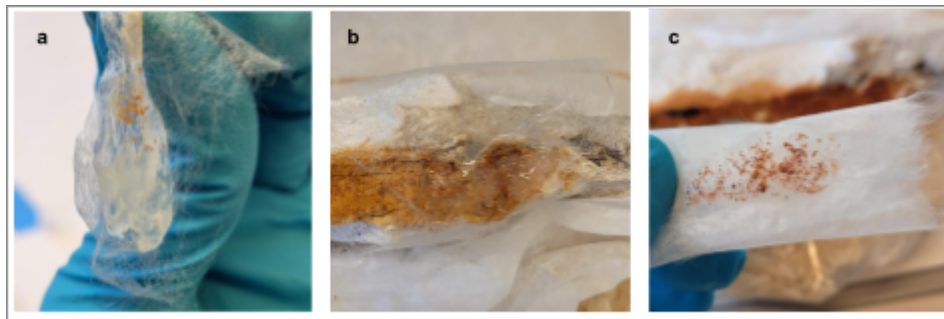
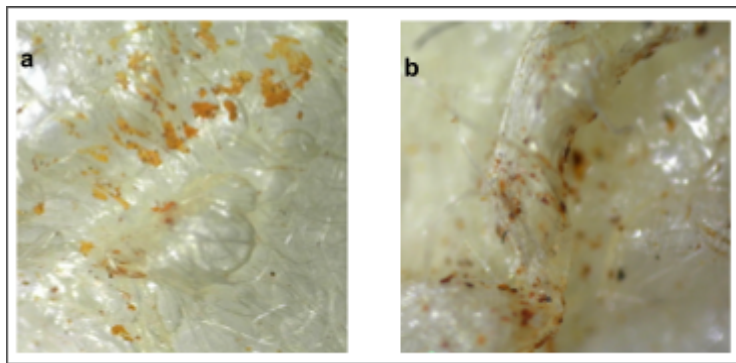


Fig. 31 - Behaviour of xanthan gel with 5oz and 10oz on sample B6. (a) JP 5oz breaking upon removal, loose fibres visible, and the weight of the gel tearing the JP. (b) Xanthan gel wetting the surface of the substrate through JP 5oz. (c) Removal of xanthan gel with 10oz JP. Note that the paper is folded in the picture, and appears sturdier than it is.

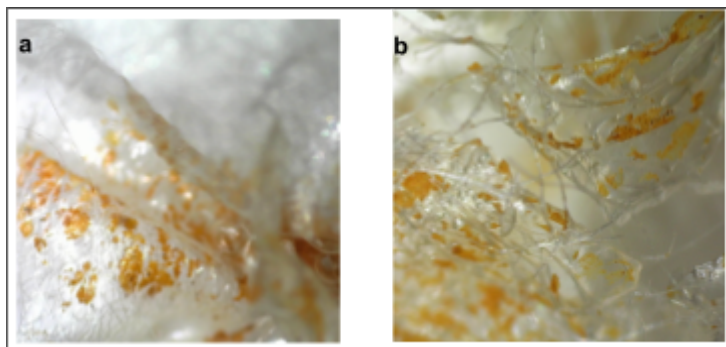
Regarding the initial gel concentration of 7% xanthan; cleaning samples show removal of corrosion products for both 2% and 5% EDTA, with slightly lower amounts assessed for 2% EDTA (fig. 32-33). Microscopy additionally indicated that with 10oz JP, lower amounts of corrosion products were removed than with 5oz regardless of EDTA concentration (fig. 33-34). This was noted most clearly when comparing cleaning samples from before and after the edited viscosity (fig. 34, 37).

Comparing results of all versions of xanthan gels; those tailored to a more appropriate, lower viscosity were able to remove corrosion products with higher efficiency.

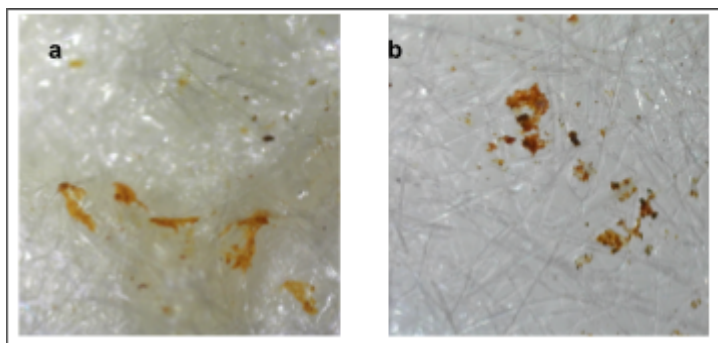
The edited viscosity gels showed a significant improvement in effect on corrosion (fig. 33-36), and the highest rate of removal was assessed for 5% EDTA, JP 10oz (fig. 37).



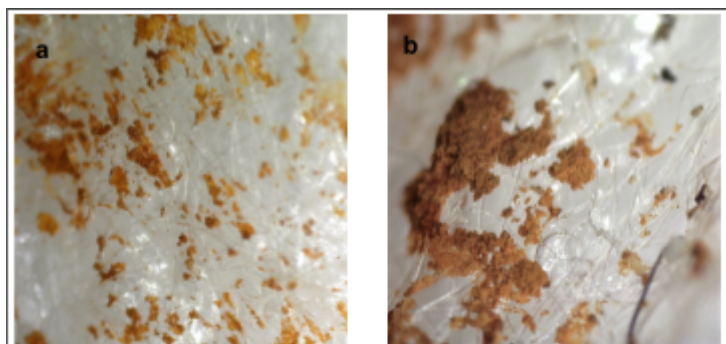
*Fig. 32 -  
Cleaning samples, fragment B3.  
7% Xanthan w. 2% EDTA, JP 5oz,  
1h.  
Magnification: (a) 61x, (b) 174x*



*Fig. 33 -  
Cleaning samples, fragment B3.  
7% Xanthan w. 5% EDTA, JP 5oz,  
1h.  
Magnification: (a) 61x, (b) 155x*

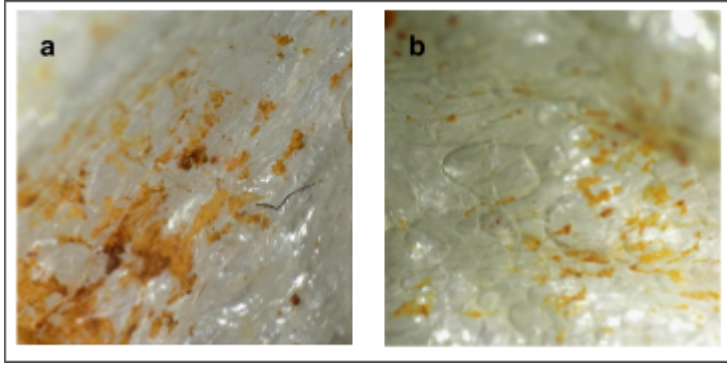


*Fig. 34 -  
Cleaning samples, fragment B3.  
(a) 7% Xanthan w. 5% EDTA, JP  
10oz, 1h.  
Magnification: 184x,  
(b) 7% Xanthan w. 2% EDTA, JP  
10oz, 1h.  
Magnification: 162x*

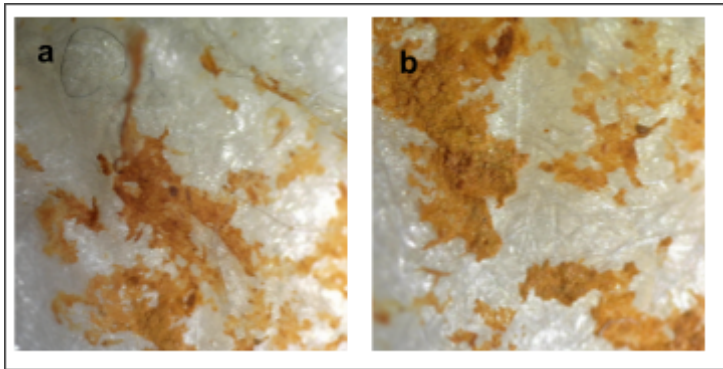


*Fig. 35 -  
Cleaning samples, fragment B6.  
3,5% Xanthan w. 2% EDTA, JP 5oz,  
1h.  
Magnification: (a) 74x, (b) 161x*





*Fig. 36 -  
Cleaning samples.  
3,5% Xanthan w. 5% EDTA, JP 5oz,  
1h.  
(a) Fragment B3.  
Magnification: 71x  
(b) Fragment B6.  
Magnification: 69x*



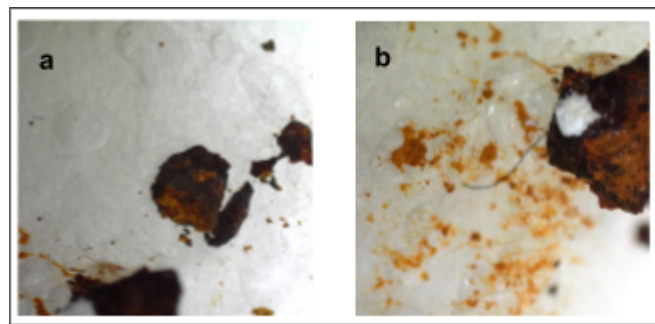
*Fig. 37 -  
Cleaning samples, fragment B3.  
3,5% Xanthan, 5% EDTA, 10oz, 1h.  
Magnification: (a) 76x (b) 159x*

3,5% Xanthan gels yielded better results regardless of JP for both 5% and 2% EDTA. The gel workability in combination with JP 5 oz still resulted in the paper breaking frequently, and subsequently involved a higher risk of residues on the substrate. Even with JP10oz, the issue remains with slight difficulty to control during application and removal.

No major fragments of plaster substrate were observed during microscopy inspection of xanthan samples. Only one of the samples showed visible fragments of plaster (*fig. 38*). In addition, samples applied to an area with armature showed fragments of iron (*fig. 39*).



*Fig. 38 - Cleaning sample, fragment B3. 3,5% Xanthan w. 2% EDTA, 5oz, 1h. Plaster particles stuck to the gel. Magnification: 67x.*

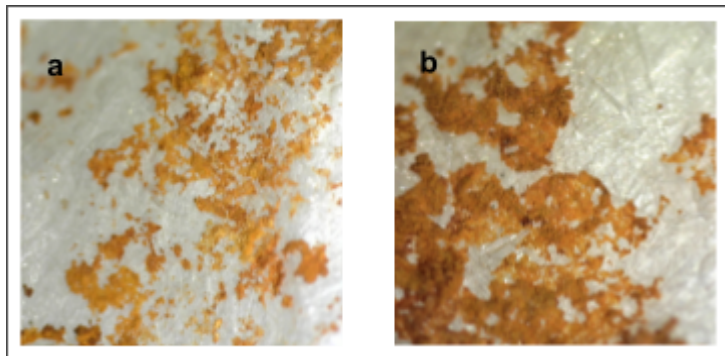


*Fig. 39 - Cleaning samples, fragment B3. 3,5% Xanthan w. 5% EDTA, 10oz, 1hr. 2nd application. Magnification: (a) 73x (b) 75x*

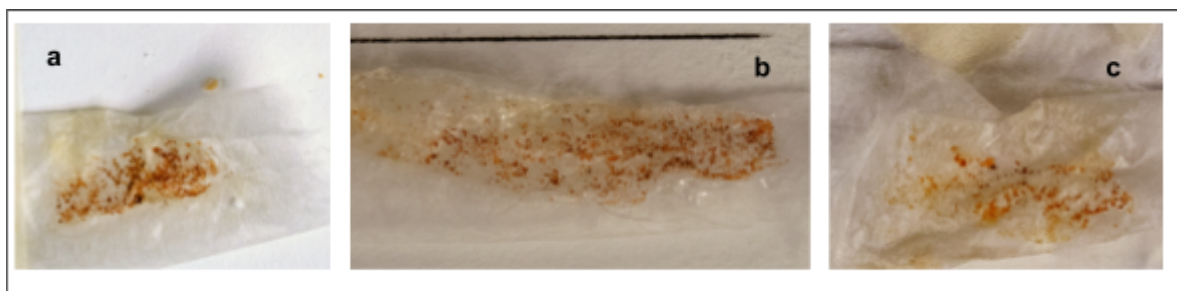
Multiple applications still resulted in corrosion product removal as can be seen when comparing cleaning samples (*fig. 37, 40-41*).

Cleaning did not result in any visible damage to the plaster, however the gels do wet the substrate more than latex and Arbocel® systems.

Overall, no significant difference was noted with the different combinations in regards to damage to the plaster substrate.



*Fig. 40*  
*Cleaning samples, fragment B6.*  
*3,5% Xanthan w. 5% EDTA,*  
*10oz, 1hr. 2nd application.*  
*Magnification:*  
*(a) 65x (b) 177x*



*Fig. 41 - Dry cleaning samples after removal. (a) 3,5% Xanthan w. 5% EDTA, 10oz, 1hr. (b) 3,5% Xanthan w. 2% EDTA, 5oz, 1hr. 2nd application. (c) 3,5% Xanthan w. 5% EDTA, 10oz, 1h, 2nd application.*

Xanthan gum behaves differently than similar gels such as Agar, during both preparation and application. Preparation alone offered some challenges due to the rapid reaction of the powder in solution. Likely more expertise with the gel itself is required to appropriately adapt concentration and time of exposure.

## 6.2 Arbocel®

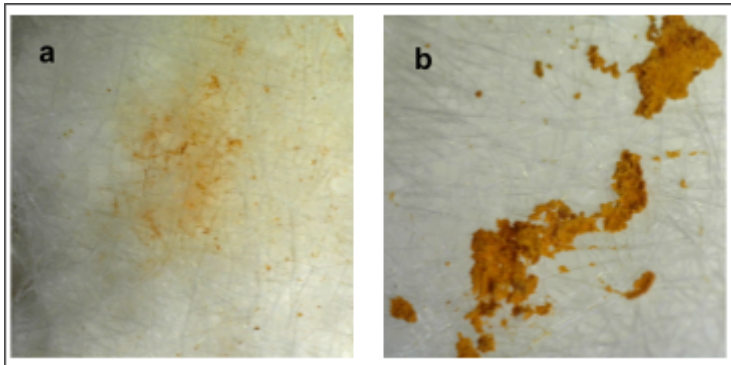
Visual examination and microscopy of Arbocel® cleaning samples indicate efficiency in removing corrosion products, at amounts similar to those of xanthan systems.

When comparing results of Arbocel® samples, results point towards higher removal rates for 5% EDTA (*fig. 42-45*). Additionally, longer exposure yielded higher rates of removal; as shown in *fig. 45-46*. 5% EDTA yielded more significant results for removal than 2% EDTA, even with shorter exposure times. Longer exposure times yielded more equal results, with only slight visible differences in favour of 5% EDTA (*fig. 43-45*).

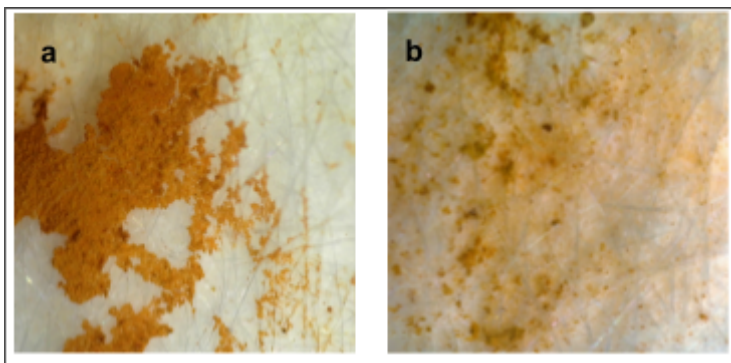
Multiple applications continued to yield results (fig. 46).

Initial testing did prove that as with xanthan gels, JP is necessary; without the tissue paper barrier the poultice became difficult to remove and left an abundance of residue. The poultices worked with both JP10oz and JP5oz, yielding similar results, albeit were easier to apply and remove with 10oz, and 10oz gave higher controllability (fig. 48).

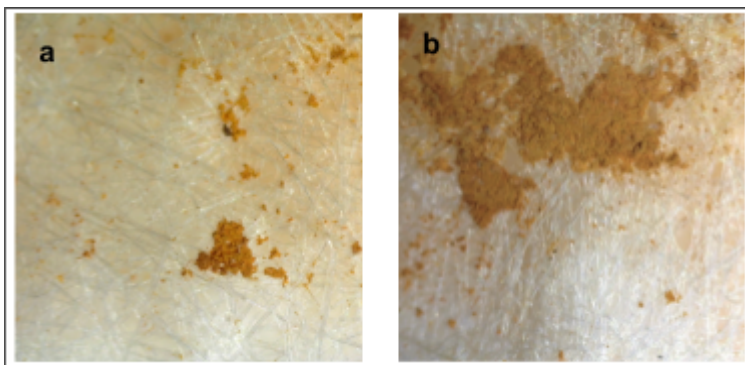
There were no visible plaster fragments on any of the cleaning samples, nor were any noted with microscopy.



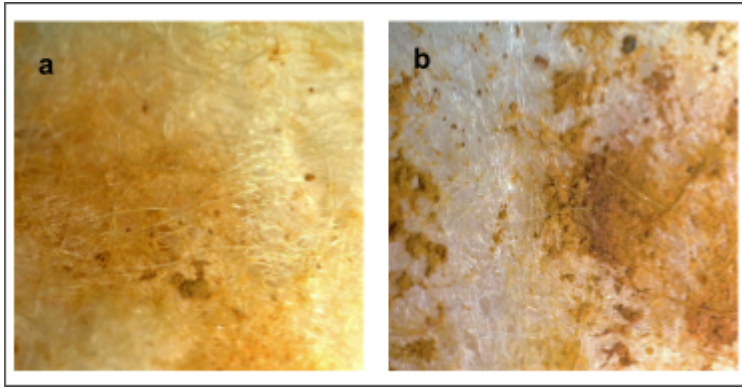
*Fig. 42-  
Cleaning samples, fragment F1.  
Arbocel®, 2% EDTA, 15-20 min,  
JP 10oz.  
Magnification: (a) 77x (b) 157x*



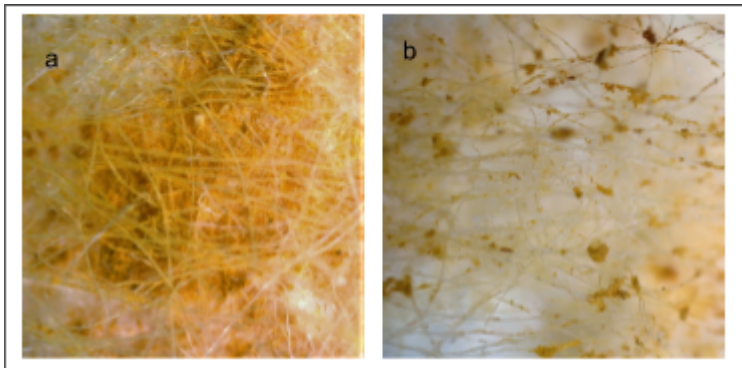
*Fig. 43 -  
Cleaning samples, fragment F1.  
Arbocel®, 2% EDTA, 5oz,  
45min.  
Magnification:  
(a) 144x (b) 141x*



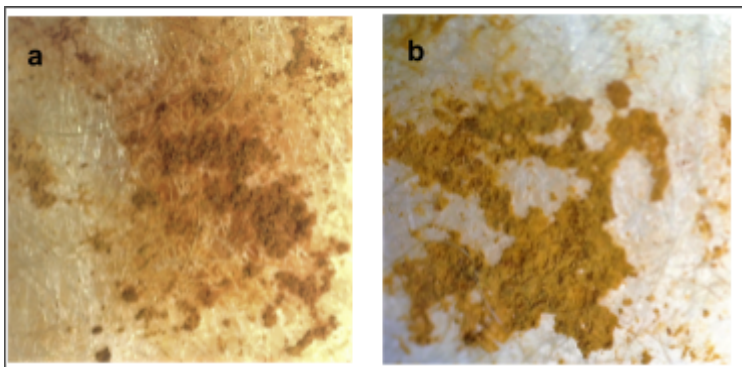
*Fig. 44 -  
Cleaning samples, fragment F1.  
Arbocel®, 5% EDTA, 10oz.  
(a) 15-20 min  
Magnification: 138x  
(b) 30min.  
Magnification: 76x*



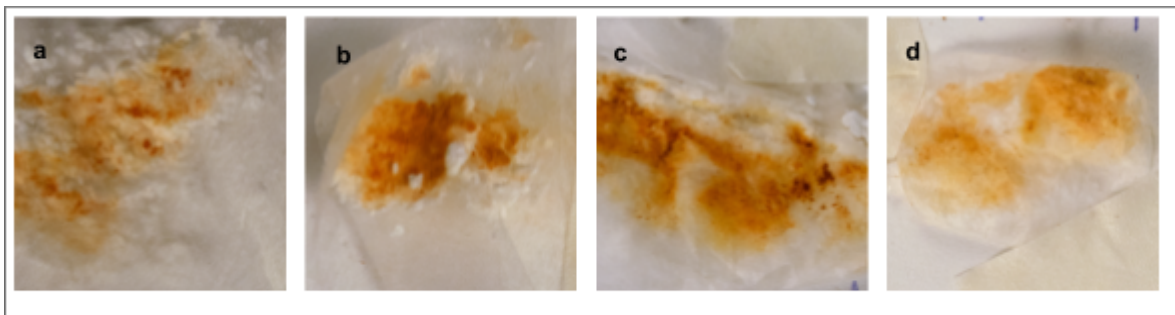
*Fig. 45 -  
Cleaning samples, fragment F1,  
Arbocel®, 5% EDTA 10oz.  
(a) 45min  
Magnification  
(b) 30min.  
Magnification: 77x*



*Fig. 46 -  
Cleaning samples, fragment F1.  
Arbocel®, 5% EDTA, 5oz.  
45min. 2nd application.  
Magnification: (a) 128x (b) 109x*

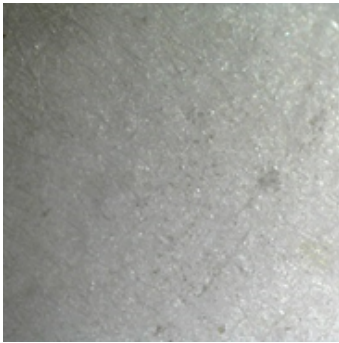


*Fig. 47 -  
Cleaning samples, fragment F1.  
Arbocel®, 5% EDTA, 10oz.  
30min, 1h, 2nd application.  
Magnification: (a) 78x (b) 85x*



*Fig. 48 - Dry Arbocel® cleaning samples. (a) 2% EDTA, 5oz, 45min. (b) 5% EDTA, 5oz, 45min.  
(c) 5% EDTA, 10oz, 30min. (d) 5% EDTA, 10oz, 45min, 3rd application.*

Arbocel® poultices left no visible residue and did not stick to the substrate. Application of Arbocel® poultice to the original cast surface yielded neither removal of corrosion nor substrate fragments; potentially due to differences in porosity (*fig. 49*). Preparation was easy to perform and could be done quickly. Poultices were easy to apply and remove, and offered good control in regards to spread and local application.



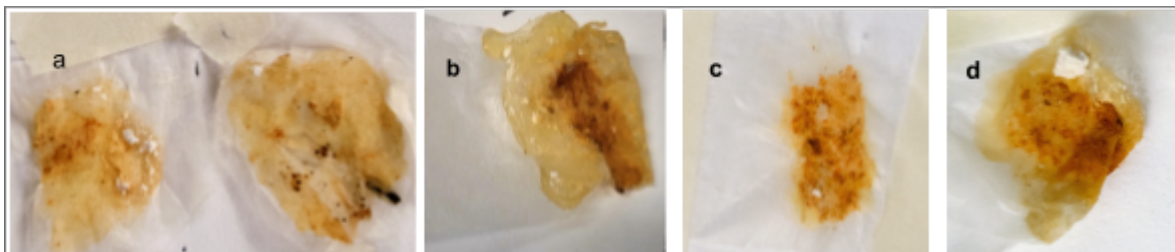
*Fig. 49 - Original cast surface cleaning sample.  
Fragment F1. Arbocel®, 5%EDTA, 10oz, 1h.  
Magnification: 77x*

### 6.3 Latex

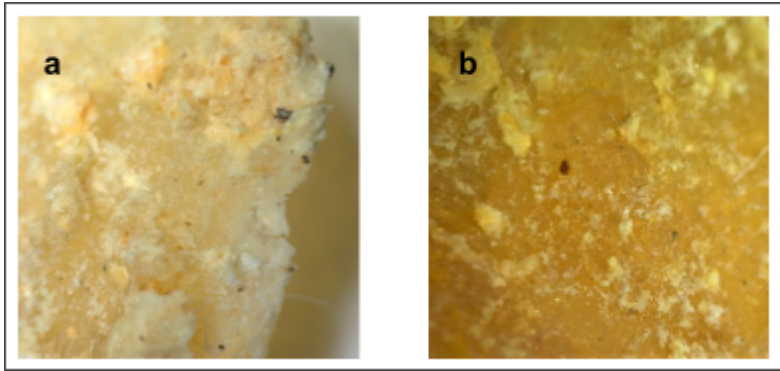
Latex systems appear to have some effect on corrosion products and their removal, though it was difficult to rate efficiency in that regard; microscopy and visual examination of all latex samples show a combination of plaster fragments and corrosion products. Overall it was difficult to tell how much of the mass removed was plaster, and how much was corrosion products (*fig. 50-52*). In addition, several samples show fragments of armature also being removed (*fig. 50, fig. 53*).

The potential damage to the substrate was significant and the extent of system influence on the plaster substrate was difficult to control.

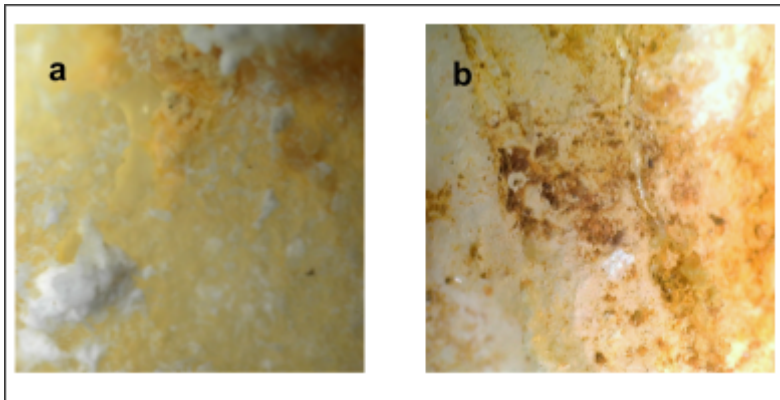
Time did influence results, both in regards to substrate influence and corrosion products removed (*fig. 50, 53, 55-56*); however even shortened exposure time involved a significant risk of removing plaster fragments.



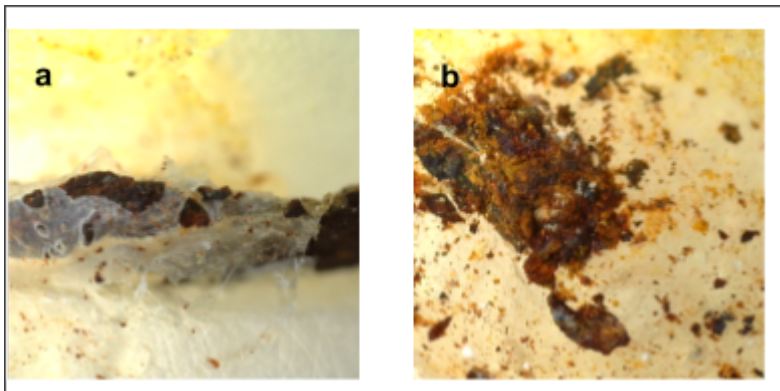
*Fig. 50 -  
Dry latex cleaning samples. Small fragments of plaster and corrosion products visible on all samples.  
(a) 2% EDTA, 10oz, 1hr, fragment B3. (b) 2% EDTA, 10oz, 1h, fragment B6  
(c) 5% EDTA, 5min, 5oz, fragment B3. (d) 5% EDTA, 30min, fragment B3.*



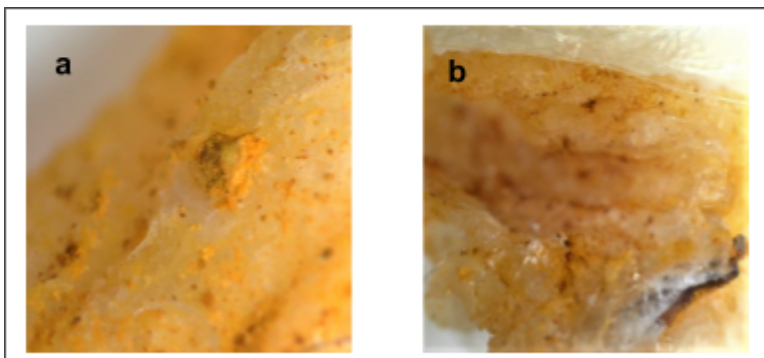
*Fig. 51 -  
Cleaning samples, latex.  
(a) 2% EDTA, 10oz, 1h,  
fragment B3.  
Magnification: 77x  
(b) 2% EDTA, 10oz, 1h,  
fragment B6.  
Magnification: 89x*



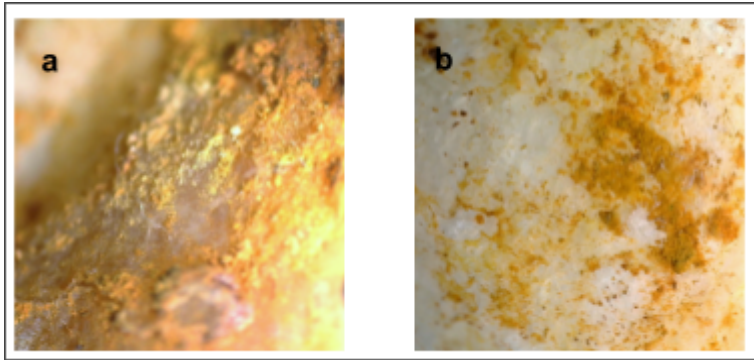
*Fig. 52 -  
Cleaning samples.  
Latex, 2% EDTA, 10oz,  
30min.  
(a) Fragment B3  
Magnification: 74x.  
(b) Fragment B6.  
Magnification: 77x*



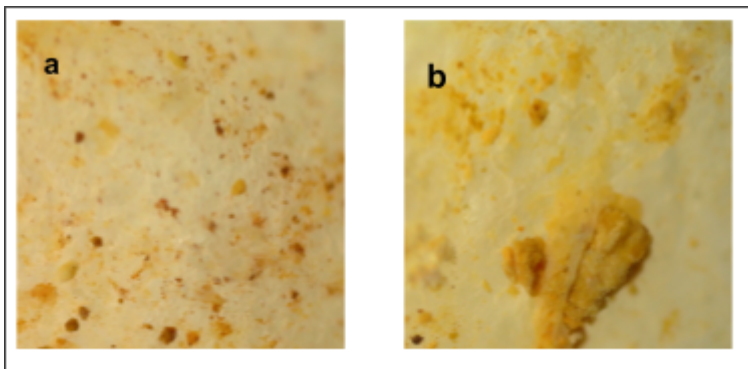
*Fig.- 53  
Cleaning samples, fragment  
B3.  
(a) Latex, 2% EDTA, 10oz,  
30min.  
Magnification: 83x  
(b) Latex, 2% EDTA, 10oz, 1h  
Magnification: 77x*



*Fig. 54 -  
Cleaning samples, fragment  
F1. Latex, 5% EDTA, 5oz,  
5min.  
(a) Magnification: 107x  
(b) Magnification: 76x*



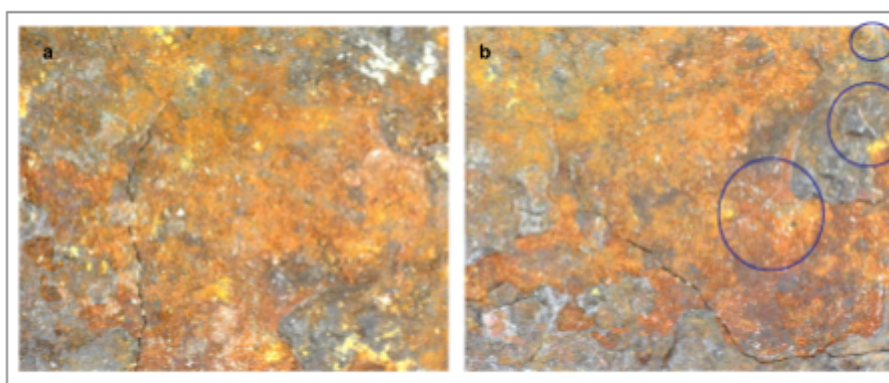
*Fig. 55 -  
Cleaning samples, fragment  
F1 .  
(a) Latex, 5% EDTA, 10oz ,  
30min.  
Magnification: 132x  
(b) Latex, 5%EDTA,10oz.  
5min.  
Magnification: 76x*



*Fig. 56 -  
Cleaning samples fragment  
B3.  
Latex, 5% EDTA, 10oz, 10min.  
Magnification: (a)-(b) 162x*

EDTA concentration did not seem to make a difference, neither for plaster removed nor corrosion products removed (*fig. 52-53, 55-56*). JP used did not influence efficiency in this regard either, albeit as with Arbocel® and xanthan systems, 5oz was suboptimal; removal and application became difficult and risk of residue was increased. Residues in this case were either bits of latex film, fibres from JP, or both.

The unevenness of the surface of the substrate in addition to the strong adherence of the latex was problematic; the latex would easily stick to microcracks, and residual latex as well as JP broke off during removal (*fig. 58*). Method also showed a higher amount of fibres from JP (*fig. 57-58*).

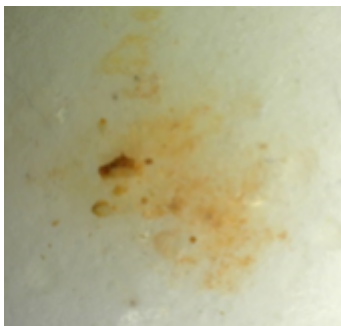


*Fig. - 57  
Microscopy of  
armature on fragment  
B3.  
(a) before cleaning  
tests.  
(b) after cleaning  
tests.  
Blue circles mark the  
location of fibres stuck  
to the surface.  
Magnification: 63x*

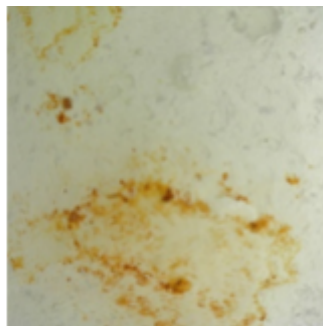


*Fig. 58 -  
Latex residue and  
fibres stuck to areas  
on B3 treated with  
latex and xanthan.  
Magnification: 60x*

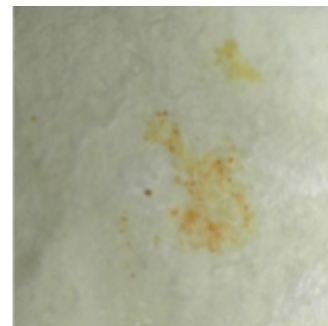
Application on the original cast surface gave clearer results than those on break edges, far more easy to interpret. As for substrate stratigraphy, the difference in results of cast surface and break edge application was likely influenced by condition and porosity of the surface. Results of external applications of latex showed a significant decrease in removal of fragments from the plaster surface (*fig. 59*). As with break edge applications, there is little to no discrepancy between 2% and 5% EDTA. The influence of time here was difficult to gauge as the variation in exposure also follows multiple applications - three in total (*fig. 59-61*). There was a visible change in corrosion staining after the first test which likely affected the outcome of the ones that followed (*fig. 30*).



*Fig. 59 -  
Cleaning sample from original cast  
surface, F1.  
Latex, 2%, 10oz, 1h  
Magnification: 73x*



*Fig. 60 -  
Cleaning sample from original  
cast surface, F1.  
Latex, 5%, 10oz, 5min  
Magnification: 163x*



*Fig. 61 -  
Cleaning sample from original  
cast surface, F1.  
Latex, 5%, 10oz, 5min  
Magnification: 74x*



Latex systems were relatively easy to use though mixing required being mindful of potential precipitate formation. Additionally, multiple batches were required due to the latex setting over time and the mixtures starting to clump. Application went without issue, and the mixture of latex systems flowed smoothly when applied with a brush. Removal was similarly without difficulties in regards to ease of use.

## 6.4 Colorimetry

Results of colorimetry calculations show what would be considered a perceptible difference for  $\Delta E$ , as in total colour change (*Table 1*).

Regarding brightness,  $\Delta L$  values indicate changes towards both lighter and darker colour. For 6 out of 11 areas  $\Delta L$  is positive, and for the remaining 5,  $\Delta L$  is negative. The majority of areas measured yielded positive values for  $\Delta a$  and  $\Delta b$  - which would indicate a shift towards yellowness-redness in the colour space (Saunders; 2020). Results of colorimetry are further elaborated upon in *6.1 Discussion and conclusion*.

Fragment and area	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta^*E$
B3 1	11,23	1,27	7,58	13,61
B3 2	9,68	-0,51	3,23	10,22
B3 3	3,16	0,06	1,16	3,37
B3 4	5,74	4,99	17,34	18,94
B3 5	3,00	4,80	9,93	11,43
B3 6	3,29	-0,01	1,54	3,63
B6 1	5,82	2,54	23,71	24,54
B6 2	-0,25	1,78	4,30	4,66
F1 1	-9,07	0,709	-0,20	9,20
F1 2	-6,54	3,63	5,36	9,14
F1 3	-6,00	3,91	7,23	10,18
F1 4	-2,73	3,50	5,66	7,19

*Table 1 - Results of calculations for  $\Delta L$ ,  $\Delta a$ ,  $\Delta b$  and  $\Delta E$  from before and after cleaning tests for select areas on fragments B3, B6 and F1. Values are rounded off to two decimals for convenience.*



Fig. 62 - Map of areas for colorimetry values.

## 6.5 XRF analysis

As noted in 5.2.2 *XRF*, XRF analysis was performed on fragments *S9* and *S11*, a piece of armature, also taken from the sculpture, and on a selection of used cleaning samples. XRF analysis of the plaster substrate, taken from both a fresh break edge and an old one, indicate the primary component as being sulphur - results of analysis from one old break edge compared to the newer one show a near equivalent percentage of sulphur (*Table 2*). In comparison, results taken from another old break edge and from an original cast surface, both show approximately a 50% division of sulphur and calcium (*Table 2*). Note that other elements present with a percentage below the error margin have been excluded here.

Element	Plaster Sample 1 New break edge	Plaster Sample 2 Old break edge	Plaster Sample 3 Old break edge	Plaster Sample 4 External
S	95,8%	47,2%	99,5%	46,5%
Ca	-	46,6%	-	53,3%

Table 2 - Results of XRF analysis of plaster substrate.

Gypsum plaster, when looking at the molecular structure and composition, contains similar amounts of sulphur and calcium. Results of analysis of plaster samples only displaying sulphur as the main component are deemed as partial errors due to the fact that the material itself is known, regardless of quantitative composition. FTIR analysis additionally confirms that the mass of the sculpture consists of plaster of paris. This is relevant when looking at results of cleaning sample analysis, which were also done with XRF. Samples were analysed in order to aid with determining substrate and corrosion product removal.

In the conclusion of this essay, sulphur present in cleaning samples is considered as part of the plaster substrate.

### Results of cleaning sample analysis

Percentages of iron, sulphur and calcium for each of the cleaning samples analysed can be found in *Table 3*. As with results of plaster sample analysis, elements with percentage below the error margin have been excluded, and complete results are included in *Appendix IV - XRF*. As previously stated, only a selection of these were analysed - due to time and resources it was not possible to analyse all. The samples selected were those thought to best represent the average of the different systems .

Element	Latex, 2% EDTA, 10oz - Plaster fragment	Latex, 2% EDTA, 10oz	3,5 Xanthan, 2% EDTA	3,5% Xanthan, 5% EDTA, 10oz	Arbocel®, 5% EDTA, 10oz
S	50,6%	47,1%	-	-	-
Ca	48,4%	30,5%	57,4%	28,8%	24,3%
Fe	-	22,4%	42,6%	68,8%	75,7%

*Tabell 3 - Results of XRF analysis of plaster substrate.*

The latex sample analysed was one used on *B6*, with 2 % EDTA and applied for 1h. Two areas on the latex sample were analysed; the first containing larger, visible plaster fragments, and the second area containing a mixture of what appeared to be both plaster and corrosion products (*see Appendix IV - XRF*).

Results from the first area confirmed that the fragments removed did indeed consist of plaster. Sulphur and calcium percentages are near equal and the amount of iron is negligible within error margins. Results of the second area show the presence of calcium, sulphur and iron, well outside error margins - indicating removal of both corrosion products and plaster substrate.

Of the xanthan samples XRF analysis was done on 3,5 % *Xanthan w. 2%EDTA, 5oz* and 3,5 % *Xanthan w. 5% EDTA, 10oz*, both used on *B6*. Results indicate an equal amount of calcium and iron present for 2% EDTA, 5oz. For 5% EDTA 10oz, the percentage of iron is significantly higher and that of calcium is much lower. This would indicate that the increased concentration of EDTA is preferable, as well as the use of 10 oz with the xanthan gel. Analysis of *Arbocel®, 5% EDTA, 10oz* show a high amount of iron present, as well as a lower amount of calcium, similar to that of 3,5 % *Xanthan, 5% EDTA, 10oz*. This indicates a comparatively above average rate of corrosion product removal, as well as a lesser degree of plaster fragment removal.

## 7. Discussion and conclusion

### 7.1 Discussion and conclusion

Whilst the original aim of this study was to determine efficiency of the tested methods, results have shown more in regards to their potential, as well as underlining the importance of the condition of the surface and its stratigraphy.

Xanthan gels showed promising results in regard to corrosion product removal when reviewing results from XRF and microscopy. XRF analysis of xanthan samples further confirmed that using JP 10oz yielded better results than 5oz both in regards to removing corrosion staining and not removing small plaster fragments from the substrate. Giraud et al (2021) and Cuvillier et al (2022) both point to the use of xanthan gel in cleaning as involving a risk of residue left on substrate surfaces. No conclusive evidence of residues were discovered during microscopy, however the gel was more difficult to apply than other systems, and made the JP break more easily - which does increase the risk of both gel residue and of JP fibres sticking to the surface of the substrate.

It could be argued here that the xanthan system itself has potential; with more experienced use and a more tailored approach this method may prove beneficial. The gel itself is non toxic which is beneficial both from user perspectives and the material of treated surfaces. The pH tolerance of xanthan gum is highly relevant if there is a desire or requirement to adjust such values to optimise chelating effects of specific ions and minimise interference with the substrate. Viscosity is crucial as results showed increased efficiency when adjusting viscosity for increased flow.

Arbocel® systems generally indicated positive results regarding both corrosion products and plaster fragments removed from the substrate via both XRF analysis and microscopy. The results between 2% EDTA and 5% EDTA poultices only differed marginally with increased time of exposure - this would suggest a lower concentration of EDTA could be utilised if desirable, and still be efficient on corrosion staining.

Preparation and application were easy and the poultice left little to no visible residue on the substrate. Additionally, Arbocel® systems showed promising results as out of the three it caused the least damage to the substrate and removed relatively high amounts of corrosion products, and proved most efficient in regards to ease of use.

Liquid latex used for moulding is commonly made from natural rubber. The products available commercially generally consist of 60 % latex, as is the case with the product used herein. Technical data and safety data sheets were not available via manufacturer - however latex tends to be mixed with other chemicals such as ammonia (Horie, 2010) and sometimes other additives (Joseph, 2013). The precipitate that occurred when mixing latex and EDTA solutions could be due to metal ions present in the natural rubber, a possibility indicated by research done on deproteinization of natural rubber (Moonprashit et al, 2017).

pH could also be a factor in this, as the product is highly alkaline and chelation with EDTA is known to be pH dependent. The extent to which the potential interactions between EDTA solutions and liquid latex influenced results cannot be confirmed at this point - in part due to limitations of this study, and due to the results of latex systems removing a significant amount of plaster fragments from the substrate overall.

XRF results in combination with microscopy did confirm that latex removed more of the plaster substrate than would be desirable when applied to break edges. The latex film also stuck to cracks and irregularities on the substrate surface, and even with careful peeling of the film it was not possible to guarantee no residue. The outcome of this method was difficult to control in that regard. Both the unintended removal of plaster fragments from the surface, and the low controllability are in line with the disadvantages to latex based systems addressed in both Mrovč and Ďoubal (2023) and Nessow (2010). Application of latex systems to the original cast surface seemingly indicated better results than break edge; there was a visible decrease of corrosion staining on the test area, and a drastic decrease of plaster fragments on cleaning samples.

In conclusion, latex and EDTA may or may not work together for removing corrosion products from plaster sculptures - however the system is not optimised for break edges with damaged surfaces and uneven stratigraphy; risk of excessive plaster fragment removal is too high and it is difficult to control the results of cleaning treatment as a whole.

Regarding the lack of results when Arbocel® systems were applied externally; on the topic of stone conservation, Kröner et al (2016) note on soluble salt transport into poultices and the relation to substrate porosity. For transport to be possible poultices need to achieve higher capillary pressure than the substrate (Kröner et al, 2016). In addition, the pore structure of the plaster substrate ought to have relevance. When considering the influence of intact casting skin and the differences in the porosity of the plaster, this could explain why Arbocel® poultices were effective in corrosion product removal when applied to break edges but not when applied to an original cast surface.

In conclusion, results of external application of latex and Arbocel® systems indicate that porosity and surface stratigraphy is relevant.

All combinations of latex and EDTA solutions removed fragments of the plaster substrate; as confirmed by both XRF analysis and microscopy of cleaning samples. It is unclear how much of the material removed was because of the systems themselves, and how much was due to substrate fragility and chelating effects of EDTA.

XRF showed calcium or sulphur as being present in all samples, with latex systems yielding the highest amounts. This was also confirmed in the amounts of plaster fragments visible through microscopy.

Regarding loss of plaster mass and fragments found on cleaning samples; it is clear that the condition of the plaster substrate does influence outcome regardless of cleaning method, as noted in Kröner et al (2016) on poultices used for salt extraction. It stands to reason that the state of the object does affect results to a significant degree.

Prior interventions such as cleaning may also influence results as well, as has been stated in previous studies (Campanella et al, 2022).

Results of all systems indicated plaster as being removed from the surface of all fragments. As previously stated, no prior cleaning was performed which makes it difficult to assess how much of the plaster found on cleaning samples consisted of loose plaster dust and how much of it was material removed directly from the substrate. With some samples like latex on break edges, removal of larger plaster fragments was evident - further confirmed by microscopy and XRF. Arbocel® systems yielded no visible removal of plaster - however XRF shows plaster as present on the analysed sample.

In theory, the buffering of EDTA solutions to values of pH 6,2 and above favour complex formation due to acid ionisation (Wolbers & Little, 2004). Results of this study indicated 5% EDTA, pH 6,2 as more effective with removal than 2% EDTA, pH 7,8: however whether this is due to the difference in concentration EDTA or the pH of solutions could not be confirmed within the context of this study.

There are mentions in several studies of EDTA having an affinity for calcium and may therefore affect the plaster substrate; these are primarily theoretical mentions albeit with significant substance in regards to the science (Doherty & Rivers, 2017; Wilks et al, 1992; Wolbers & Little, 2004; Wolbers, 2003). There is little evidence of this occurring in practice, though it has been confirmed by Stancliffe et al (2005) – it is a clear possibility and one that ought to be considered in relation to the results presented herein.

No significant difference was noted for plaster substrate removal with regards to EDTA concentration and pH that cannot be related to the grade of JP used. The use of JP as a barrier was necessary for all three carriers in order to minimise risk of residues. 10oz proved more efficient than 5oz when looking at all parameters, as JP 5 oz involved a significant risk of leaving fibres stuck to substrate. Percentage of calcium and iron for Arbocel® and xanthan systems were of comparatively more ideal levels when using JP10oz - it stands to reason that the use of a barrier also influenced plaster removal.

Regardless of the system, multiple applications would likely be necessary. This would apply regardless of the extent of corrosion product migration and substrate condition as is indicated by results when comparing break edge and original cast surface areas.

Regarding positive values for  $\Delta L$  - a brighter colour could indicate a decrease in corrosion products present (Cuvillier et al, 2022). Changes in  $a$  and  $b$  values indicate a change in the red-green and yellow-blue space; negative values for  $\Delta a$  and  $\Delta b$  would signify yellowing and a decrease of redness-yellowness (Saunders, 2020). Cuvillier et al (2022) attributes lower values for  $a$  and  $b$  and the shift towards blue and green to removal of rust and corrosion products.

The results from XRF analysis and microscopy of cleaning samples stand in slight contrast to the results of colorimetry; and as such both margins of error and potential causes need to be considered.

The colour variation on corrosion stained areas is distinct, and the presence of smaller armature fragments may be of relevance – these areas are visibly darker and could influence values significantly. Additionally, value differences could be related to lighting and difficulty in acquiring measurements in the same area; the surfaces of the fragments are uneven, and the fragments themselves needed to be balanced and supported whilst taking measurements. Other causes could be explained as substrate-system interactions that were not accounted for, such as further migration of corrosion products into the substrate due to wetting, or residues from cleaning systems that went unnoticed.

Regarding colorimetry, results thereof and the potential error margins; as a method for evaluating cleaning system efficiency on corrosion stains within the context of this study, it provided no conclusive data. It may be possible in future research to bypass error margins, by taking the unevenness of surface and colour variations into account in considering how that could influence results.

Visual examination and microscopy in order to evaluate effect on corrosion stains and plaster removal combined with XRF analysis proved more reliable. XRF analysis did in this case confirm results of microscopy, however the opposite could also have been clarified by the data received.

Due to the limited time and resources available, it would likely have been beneficial to perform XRF analysis of test surfaces on all fragments before and after as well as all viable cleaning samples as these methods combined provided more conclusive data than colorimetry values. And as noted in Shackley (2011), error margins for XRF can be minimised with proper preparation thus reducing the risk of line overlap and similar issues.

Preliminary tests of cleaning systems were performed on fragments not part of the main study. These tests were performed in order to evaluate carrier interactions with the plaster surface - not the effect of cleaning systems on corrosion staining. The extent of influence surface porosity, stratigraphy and condition had on results was not taken into account. Fragments utilised in the main experiments provided a limited amount of areas for testing; it would likely have benefited the study, and provided more conclusive data if preliminary tests had been performed on fragments with corrosion staining and surfaces closer to that of the break edges on those used in the experiments. The majority of tested areas on fragments *B3*, *B6* and *F1* were break edges originating from where the plaster cracked due to armature expansion, and as such these areas were more fragile and uneven than those of fragments used for preliminary testing. Corrosion migration and exposure to the elements whilst in storage may have also eroded the surfaces and made them more sensitive to cleaning. Only one of the tested fragments featured corrosion staining on the original cast surface - with consideration given to surface properties, it may have been possible to acquire additional fragments with similar damage, which would have provided more conclusive data.

### *Ethical aspects*

Some of the original material removed from the plaster substrate could be negligible due to the nature of the damaged areas; if the sculpture was to be restored some original material, including the majority of the armature itself would need to be removed and replaced. Whilst minimal intervention may be the ideal, preserving object integrity through aesthetics and historical values may in this case be more relevant than preserving original material. Particularly when treating break edges, in cases such as *Idun* it could be argued that the object's integrity is not lost as the loss of material would not interfere with the intended presentation of the sculpture itself. Additionally it would likely be more damaging to leave the sculpture in its current condition. Due to the uncertain future of *Idun*, removing as much of the corrosion as possible would be ideal. If reconstructed and retouched to cover the stains – with changing conditions, corrosion products could migrate further and cause further damage.

## 7.2 Future research

Future research into the use of chelating agents as a method for treating corrosion related damages such as those found with *Idun* ought to aim for tailored systems and potentially the use of more appropriate chelating agents with a lesser affinity for calcium.

There are also interesting alternatives to typical chelates like EDTA and HEDTA - in Campanella et al (2022) certain proteins with a high affinity for iron are investigated in relation to rust stains with promising results. Both Arbocel® and xanthan systems could be tailored to a more viable system for plaster, such as mixing different variations together with other components as is mentioned in Mrovč & Ďoubal (2023); referring to a recent study on treating corrosion in plaster casts with Arbocel® BC1000 and Kelzan® HP-T.

Additionally, the influence of surface stratigraphy when choosing a carrier, and the relevance of porosity in relation to extent of damage could be further investigated. Cleaning systems could be adapted to function efficiency on corrosion staining depending on targeted areas and whether or not they are located on original cast surfaces or break edges.

Lastly, continued discussions surrounding ethics and conservation treatments are always crucial - particularly when dealing with objects that are as complex as plaster casts.



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## Photographic documentation and illustrations

All photographic documentation done by the author, Gabriella Zimmerman, unless otherwise specified in relation to images.

Illustrations done by Gabriella Zimmerman.

## APPENDIX I – Equipment

<b>Photography</b>	
NIKON D3300	Samsung Galaxy S20 FE 5G
<b>Photo editing &amp; Illustrations</b>	
Adobe Photoshop 2023. # 24.x	Wacom Intuos Pro Paper Edition
<b>Microscopy</b>	
Dino-Lite Edge 3.0 Model: AM73915MZT Resolution: 5M pixels (2560x1920= Magnification: 10x-220x	DinoCapture 2.0
<b>FTIR</b>	
Bruker Alpha-II, Platinum-ATR	OPUS
<b>XRF</b>	
Bruker Elio Micro-XRF Spectrometer	XGLab. X and Gamma Ray Electronics.

<b>Colour measurement</b>	
Equipment	Konica Minolta CM-26d Spectrophotometer
Illumination/viewing system	Diffused illumination, 8-10°
Wavelength range	360 to 740nm
Measurement diameter	3 mm
Daylight illuminant	D65

## APPENDIX II – Cleaning tests

*Variant* indicates which version of each system was used. Selected areas are shown in *fig. 24*. Applications are noted where multiple applications of the same variant, time and JP were used. *Area* indicates break edge, *BE*, or original cast surface, *OSC*.

<b>Variant</b>	<b>Fragment</b>	<b>JP oz</b>	<b>Area</b>	<b>Time</b>	<b>Applications</b>
<i>7% Xanthan, 5% EDTA</i>	B3	10oz	<i>BE</i>	1h	-
<i>7% Xanthan, 5% EDTA</i>	F1	5oz	<i>BE</i>	1h	-
<i>7% Xanthan, 2% EDTA</i>	B3	10oz	<i>BE</i>	1h	-
<i>7% Xanthan, 2% EDTA</i>	B3	5oz	<i>BE</i>	1h	-
<i>3,5% Xanthan, 5% EDTA</i>	B3	10oz	<i>BE</i>	1h	2
<i>3,5% Xanthan, 5% EDTA</i>	B3	10oz	<i>BE</i>	1h	3
<i>3,5% Xanthan, 5% EDTA</i>	B6	10oz	<i>BE</i>	1h	2
<i>3,5% Xanthan, 5% EDTA</i>	B6	10oz	<i>BE</i>	1h	2
<i>3,5% Xanthan, 5% EDTA</i>	B3	5oz	<i>BE</i>	1h	
<i>3,5% Xanthan, 5% EDTA</i>	B6	5oz	<i>BE</i>	1h	
<i>3,5% Xanthan, 2% EDTA</i>	B3	10oz	<i>BE</i>	1h	
<i>3,5% Xanthan, 2% EDTA</i>	B3	5oz	<i>BE</i>	1h	
<i>3,5% Xanthan, 2% EDTA</i>	B6	5oz	<i>BE</i>	1h	2

*Table 4 - Cleaning tests, xanthan gels.*



<b>Variant</b>	<b>Fragment</b>	<b>JP oz</b>	<b>Time</b>	<b>Area</b>	<b>Applications</b>
<i>Latex, 2% EDTA</i>	B3	10oz	1h	<i>BE</i>	2
<i>Latex, 2% EDTA</i>	B3	10oz	30min	<i>BE</i>	2
<i>Latex 2% EDTA</i>	B3	5oz	3min	<i>BE</i>	
<i>Latex, 2% EDTA</i>	B6	10oz	1h	<i>BE</i>	
<i>Latex, 2% EDTA</i>	B6	10 oz	30min	<i>BE</i>	
<i>Latex, 2% EDTA</i>	B6	10oz	5min	<i>BE</i>	
<i>Latex, 2% EDTA</i>	F1	10oz	1h	<i>OCS</i>	
<i>Latex, 5% EDTA</i>	B3	10oz	10min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	B3	10oz	3-5 min	<i>BE</i>	2
<i>Latex, 5% EDTA</i>	B6	5oz	5min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	B6	10oz	3min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	F1	10z	30 min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	F1	10oz	5min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	F1	5oz	5min	<i>BE</i>	
<i>Latex, 5% EDTA</i>	F1	10oz	20min	<i>OCS</i>	
<i>Latex, 5% EDTA</i>	F1	10oz	5min	<i>OCS</i>	2

*Table 5 - Cleaning tests, latex systems.*

<b>Variant</b>	<b>Fragment</b>	<b>JP oz</b>	<b>Time</b>	<b>Area</b>	<b>Applications</b>
<i>Arbocel®</i> , 2% EDTA	F1	10oz	20min	<i>BE</i>	
<i>Arbocel®</i> , 2% EDTA	F1	10oz	15min	<i>BE</i>	
<i>Arbocel®</i> , 2% EDTA	F1	10oz	45min	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	5oz	45min	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	10oz	20min	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	10oz	15min	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	10oz	1h	<i>BE</i>	3
<i>Arbocel®</i> , 5% EDTA	F1	5oz	1h	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	10oz	30min	<i>BE</i>	
<i>Arbocel®</i> , 5% EDTA	F1	10oz	1h	<i>OCS</i>	

*Table 6 - Cleaning tests, Arbocel® poultices.*

## APPENDIX III – Colorimetry values

Fragment/Area	L*	a*	b*	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta E^*$
Preclean B3 Rust 1	55,51112251	13,61133792	31,71990257				
Postclean B3 Rust 1	66,74425476	14,88576861	39,29737783				
				11,23313225	1,274430692	7,577475258	13,60975992
Preclean B3 Rust 2	46,12336722	13,19729317	30,24116774				
Postclean B3 Rust 2	55,80416506	12,68821039	33,46858003				
				9,68079784	-0,509082777	3,227412285	10,21729916
Preclean B3 Rust 3	67,04949112	11,57324384	30,78761947				
Postclean B3 Rust 3	70,21140889	11,63582594	31,95207011				
				3,161917776	0,062582103	1,164450637	3,370101754
Preclean B3 Rust 4	49,85725121	15,17229834	25,79735397				
Postclean B3 Rust 4	55,59597942	20,1640022	43,14202605				
				5,738728207	4,991703867	17,34467207	18,93905378
Preclean B3 Rust 5	38,48872496	14,02783527	24,97602924				
Postclean B3 Rust 5	41,48485793	18,8220808	34,90979199				
				2,996132968	4,79424553	9,933762747	11,42984013
Preclean B3 Rust 6	57,78640097	12,22456528	31,57018598				
Postclean B3 Rust 6	61,07762977	12,21222339	33,11308187				
				3,291228795	-0,012341888	1,542895889	3,63495076
Preclean B6 Rust 1	55,95494176	10,20835989	26,8011904				
Postclean B6 Rust 1	61,77627131	12,74887781	33,9214318				
				5,821329548	2,540517919	23,71307191	24,54896919
Preclean B6 Rust 2	61,40648959	13,02725317	33,50998668				
Postclean B6 Rust 2	61,16124793	14,81070969	37,81001848				
				-0,24524166	1,783456522	4,300031802	4,661666455
Preclean F1 Rust 1	45,86386769	19,31977986	35,36290689				
Postclean F1 Rust 1	36,79613397	20,02900728	35,16695079				
				-9,067733722	0,709227426	-0,195956094	9,097537974
Preclean F1 Rust 2	48,18299233	19,33485967	33,31799658				
Postclean F1 Rust 2	41,72970631	22,96813858	38,68248134				
				-6,453286019	3,633278908	5,364484762	9,14457833
Preclean F1 Rust 3	44,22283445	15,36857853	24,13576037				
Postclean F1 Rust 13	38,22004141	19,27953997	31,36191581				
				-6,002793039	3,910961434	7,226155442	10,1757784
Preclean F1 Rust 4	52,57598537	17,96437381	34,59615551				
Postclean F1 Rust 4	49,84451659	21,46233719	40,25922954				
				-2,731468778	3,497963381	5,663074036	7,194934123

Table 6 - Results of colorimetry measurements, full values included.

# APPENDIX IV - XRF

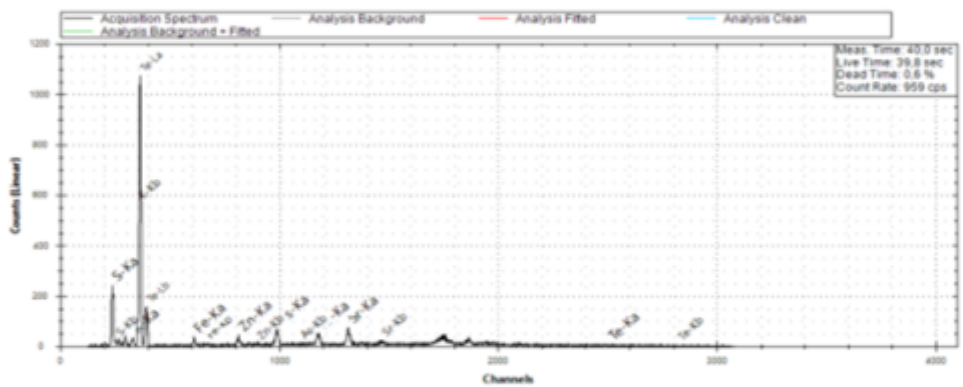
## XRF - IDUN - Plaster, new break edge



13/04/2023 16:45:35

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air

### Spectrum:



### Analysis Results:

Element	Concentration	Error
S	94,79%	±2,86%
K	4,05%	±10,73%
Fe	0,28%	±7,51%
As	0,27%	±4,27%
Kr	0,18%	±4,87%
Zn	0,17%	±6,41%
Sr	0,17%	±4,58%
Te	0,09%	±1,28%

Analysis Date and Time: 13/04/2023 16:41:42  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

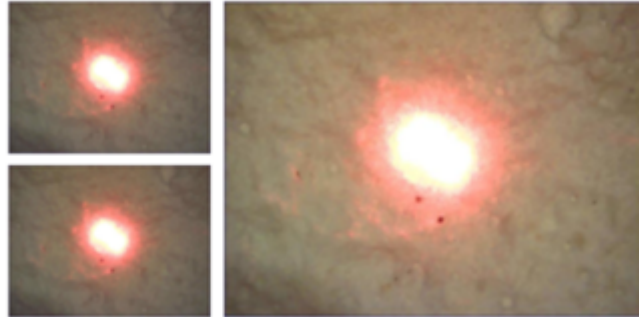
Excluded Elements for Fitting Analysis:  
 Ar, Rh

# XRF - IDUN - Plaster, Old break edge

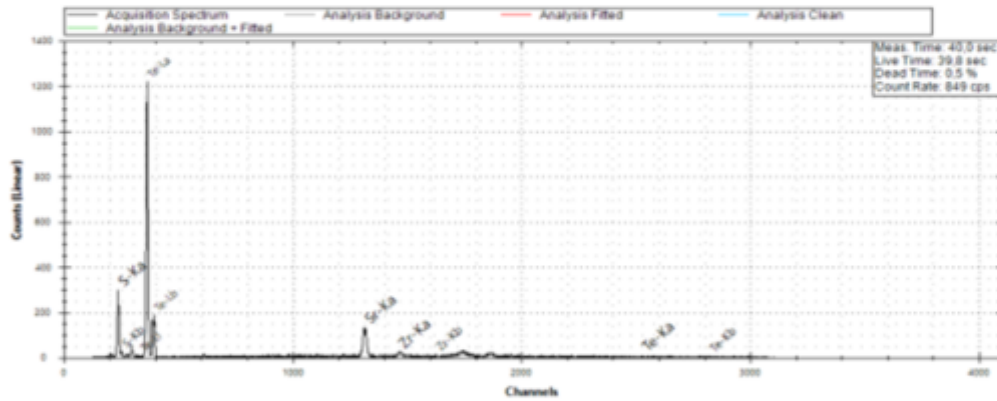


13/04/2023 16:04:10

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



## Spectrum:



## Analysis Results:

Element	Concentration	Error
S	99,47%	±2,17%
Sr	0,44%	±2,21%
Te	0,07%	±1%
Zr	0,01%	±9,95%

Analysis Date and Time: 13/04/2023 16:03:15  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

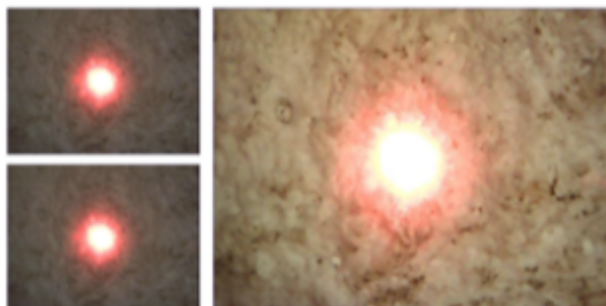
Excluded Elements for Fitting Analysis:  
 Ar, Rh

# XRF - IDUN - Plaster, Old break edge 2nd

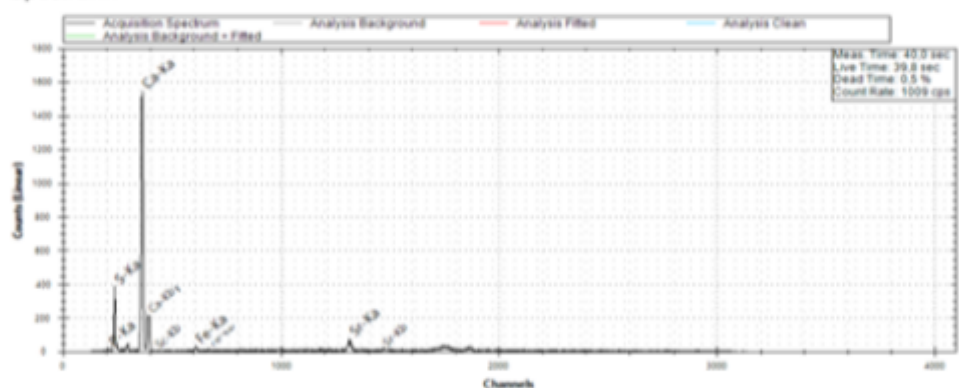


13/04/2023 16:11:25

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



## Spectrum:



## Analysis Results:

Element	Concentration	Error
S	47,19%	±1,99%
Ca	46,93%	±0,85%
P	3,27%	±7,12%
Sc	2,43%	±3,16%
Fe	0,11%	±5,92%
Sr	0,07%	±3,48%

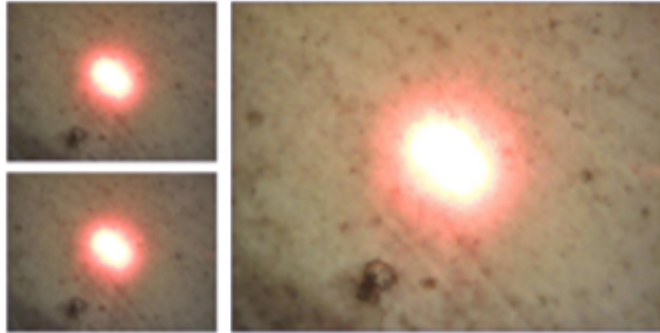
Analysis Date and Time: 13/04/2023 16:10:45  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False  
 Excluded Elements for Fitting Analysis:  
 Ar, Rh

# XRF - IDUN - Plaster, Cast Surface

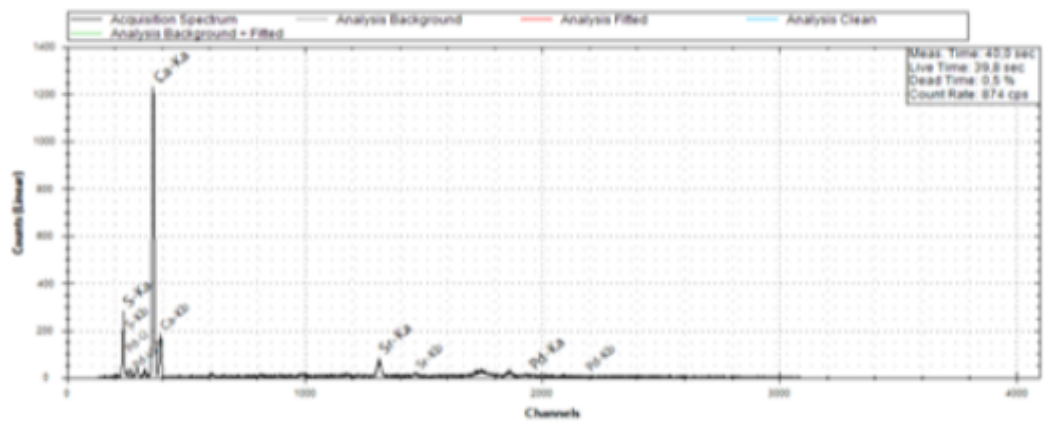


13/04/2023 15:53:28

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elilo Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



## Spectrum:



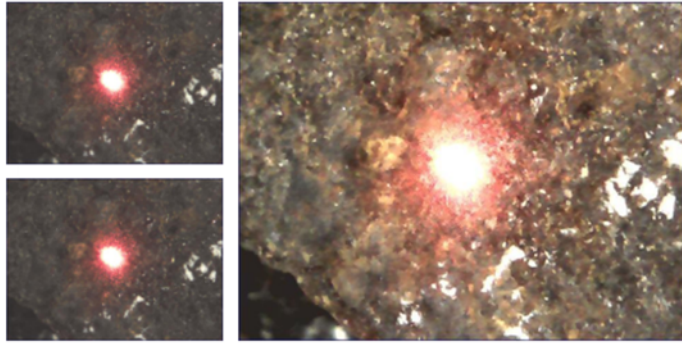
## Analysis Results:

Element	Concentration	Error
Ca	53,33%	±1,03%
S	46,48%	±2,49%
Sr	0,14%	±3,31%
Pd	0,05%	±5,4%

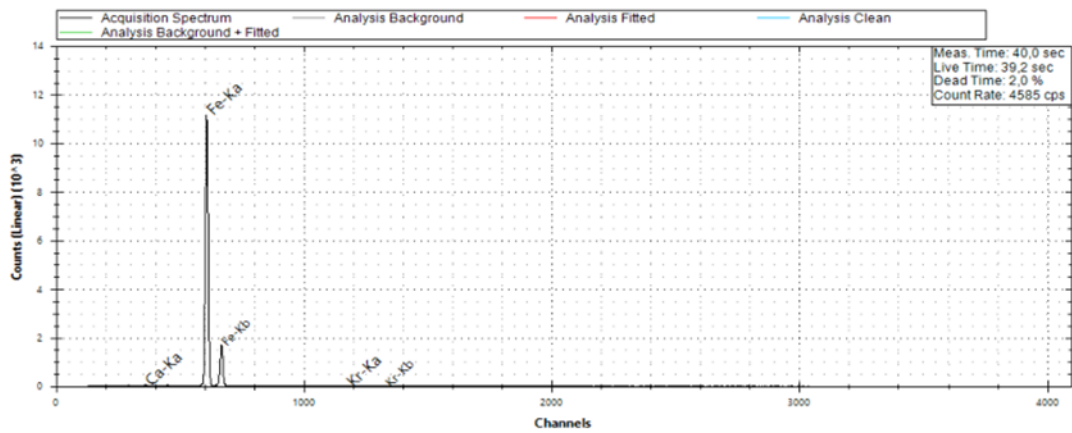
Analysis Date and Time: 13/04/2023 15:52:46  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

Excluded Elements for Fitting Analysis:  
 Ar, Rh

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



Spectrum:



Analysis Results:

Element	Concentration	Error
Fe	97,73%	±0,27%
Ca	2,11%	±4,67%
Kr	0,16%	±6,82%

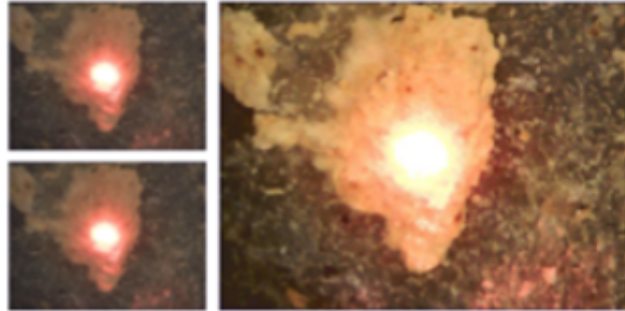
Analysis Date and Time: 13/04/2023 15:41:22  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

Excluded Elements for Fitting Analysis:  
 Ar, Rh

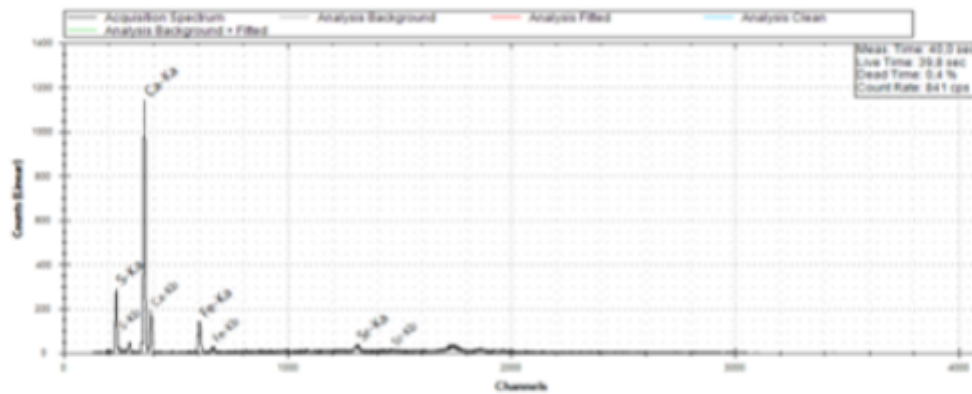


03/05/2023 13:17:44

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



**Spectrum:**



**Analysis Results:**

Element	Concentration	Error
Ca	50,69%	±0,97%
S	48,39%	±2,22%
Fe	0,85%	±2,55%
Sr	0,06%	±4,49%

Analysis Date and Time: 03/05/2023 13:17:29  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

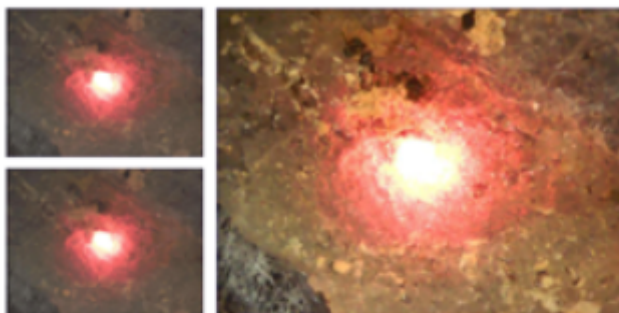
Excluded Elements for Fitting Analysis:  
 Kr, Ar, Rh

IDUN - Cleaning sample, B6, Latex 2% EDTA, 1h

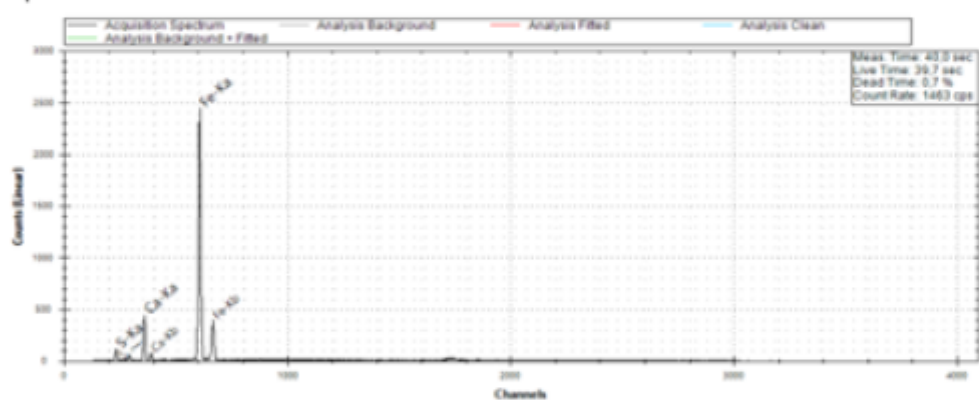


03/05/2023 13:20:41

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



Spectrum:



Analysis Results:

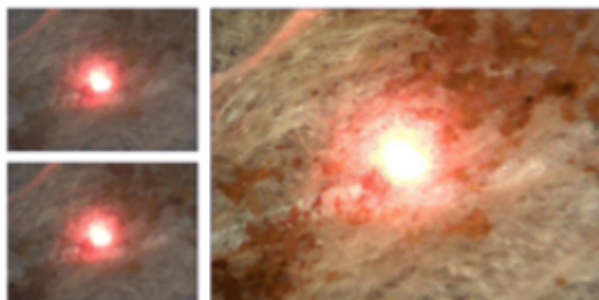
Element	Concentration	Error
S	47,1%	±3,08%
Ca	30,49%	±1,53%
Fe	22,41%	±0,58%

Analysis Date and Time: 03/05/2023 13:20:29  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

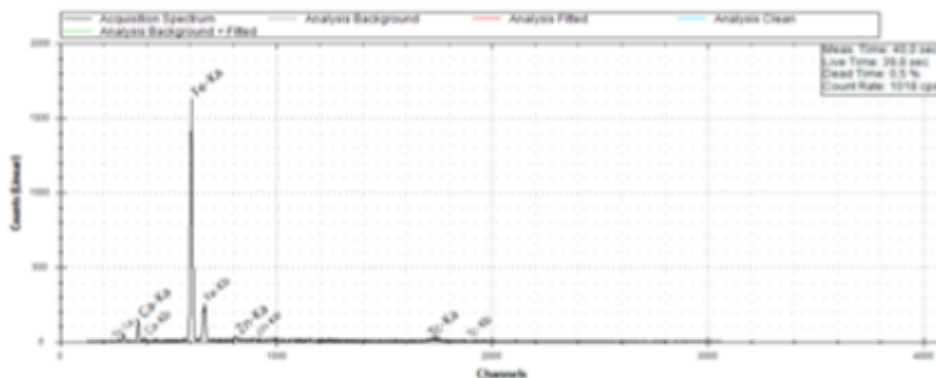
Excluded Elements for Fitting Analysis:  
 Kr, Ar, Rh

03/05/2023 13:27:13

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



Spectrum:



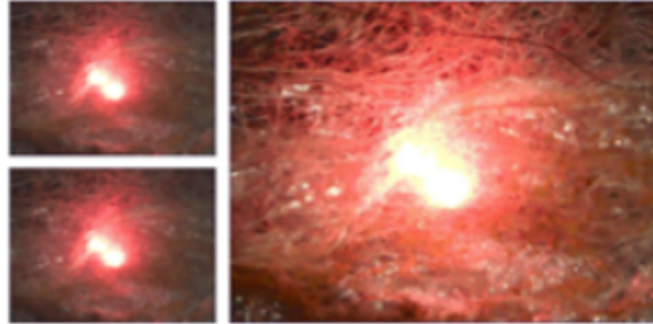
Analysis Results:

Element	Concentration	Error
Fe	68,83%	±0,75%
Ca	28,78%	±2,74%
Zn	1,44%	±5,49%
Tc	0,95%	±6,54%

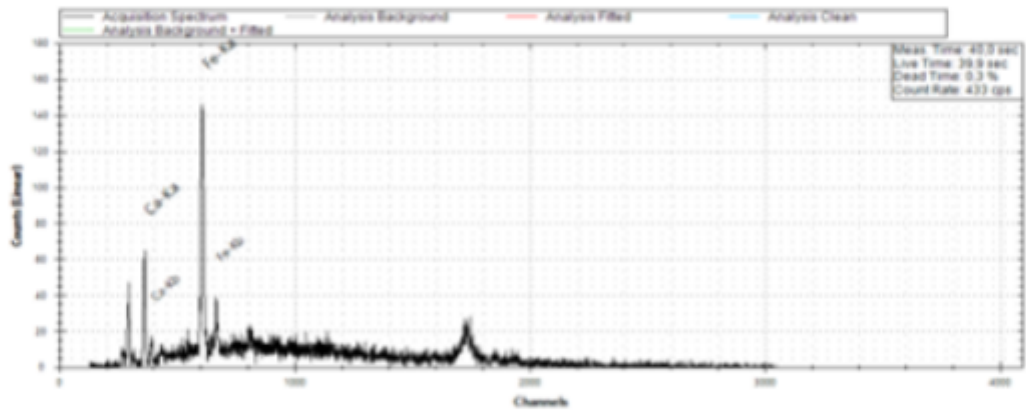
Analysis Date and Time: 03/05/2023 13:26:57  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

Excluded Elements for Fitting Analysis:  
 Kr, Ar, Rh

Measurement Time: 40,0 s  
Tube Voltage: 40 kV  
Tube Current: 20 µA  
Tube Target Material: Rh  
Elio Device: SN1253  
Device Mode: Head  
Acquisition Mode: Manual  
Acquisition Channels: 4096  
Sample to Detector Material: Air



**Spectrum:**



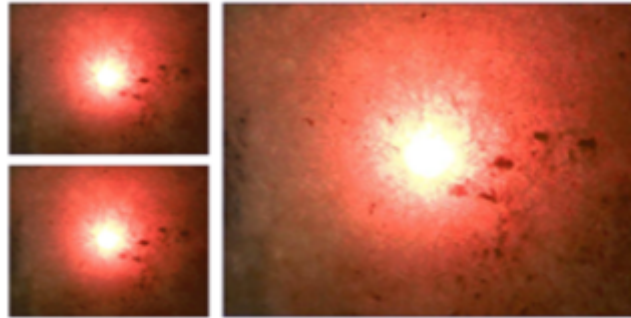
**Analysis Results:**

Element	Concentration	Error
Ca	57,39%	±4,41%
Fe	42,61%	±2,39%

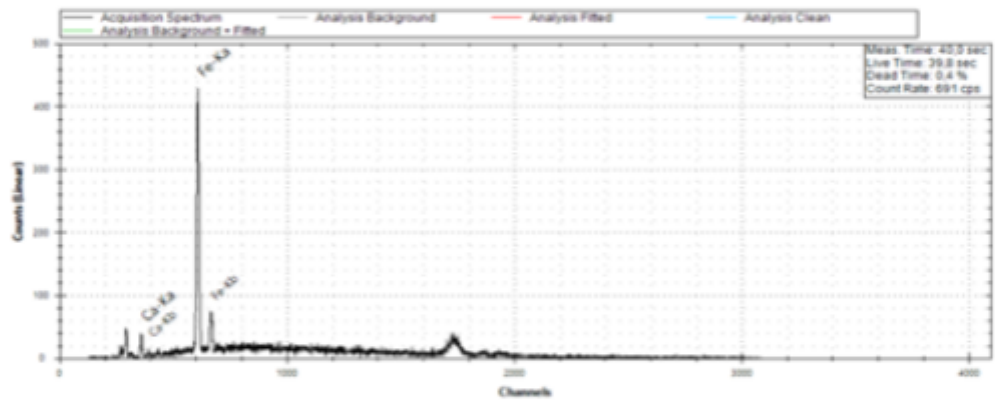
Analysis Date and Time: 03/05/2023 13:23:44  
Analysis Type: Automatic  
Spectrum Left Cut: 1 keV  
Spectrum Right Cut: 50 keV  
Spectrum Upper Limit: 50 keV  
Use M Line: False  
Super Impose Peak Areas: False

Excluded Elements for Fitting Analysis:  
Kr, Ar, Rh

Measurement Time: 40,0 s  
 Tube Voltage: 40 kV  
 Tube Current: 20 µA  
 Tube Target Material: Rh  
 Elio Device: SN1253  
 Device Mode: Head  
 Acquisition Mode: Manual  
 Acquisition Channels: 4096  
 Sample to Detector Material: Air



**Spectrum:**



**Analysis Results:**

Element	Concentration	Error
Fe	75,66%	±1,41%
Ca	24,34%	±5,85%

Analysis Date and Time: 03/05/2023 13:29:33  
 Analysis Type: Automatic  
 Spectrum Left Cut: 1 keV  
 Spectrum Right Cut: 50 keV  
 Spectrum Upper Limit: 50 keV  
 Use M Line: False  
 Super Impose Peak Areas: False

Excluded Elements for Fitting Analysis:  
 Kr, Ar, Rh

## APPENDIX V - FTIR

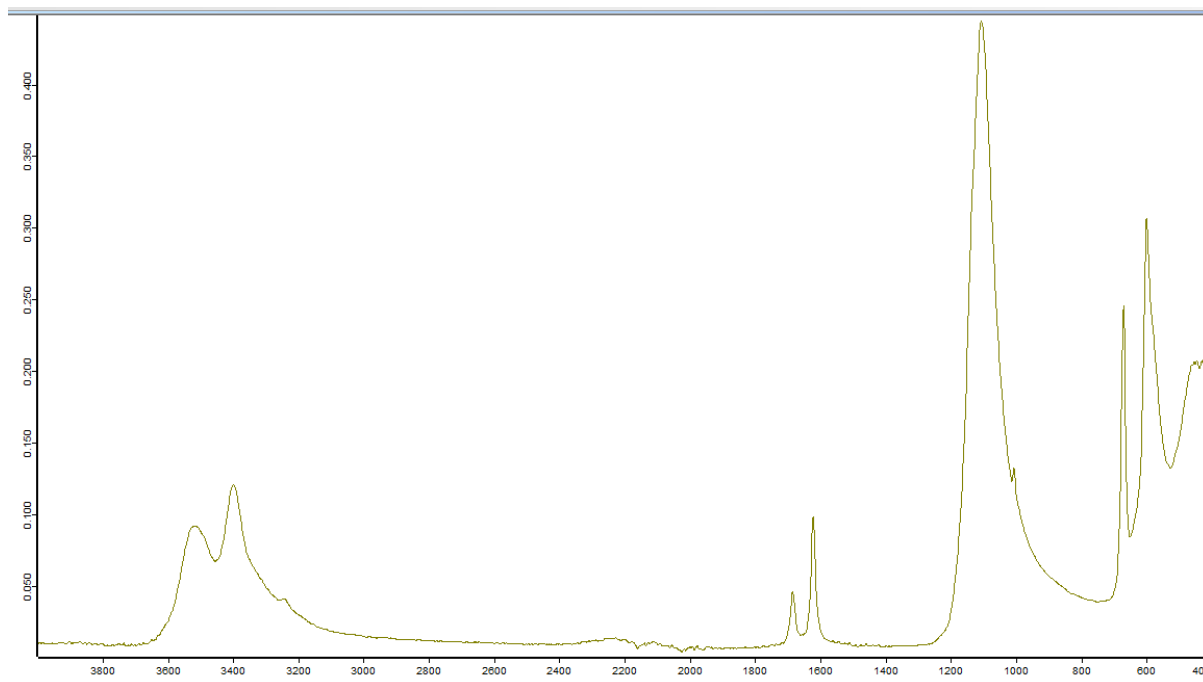


Fig. 63 - FTIR Spectrum. Plaster sample, fragment S9.

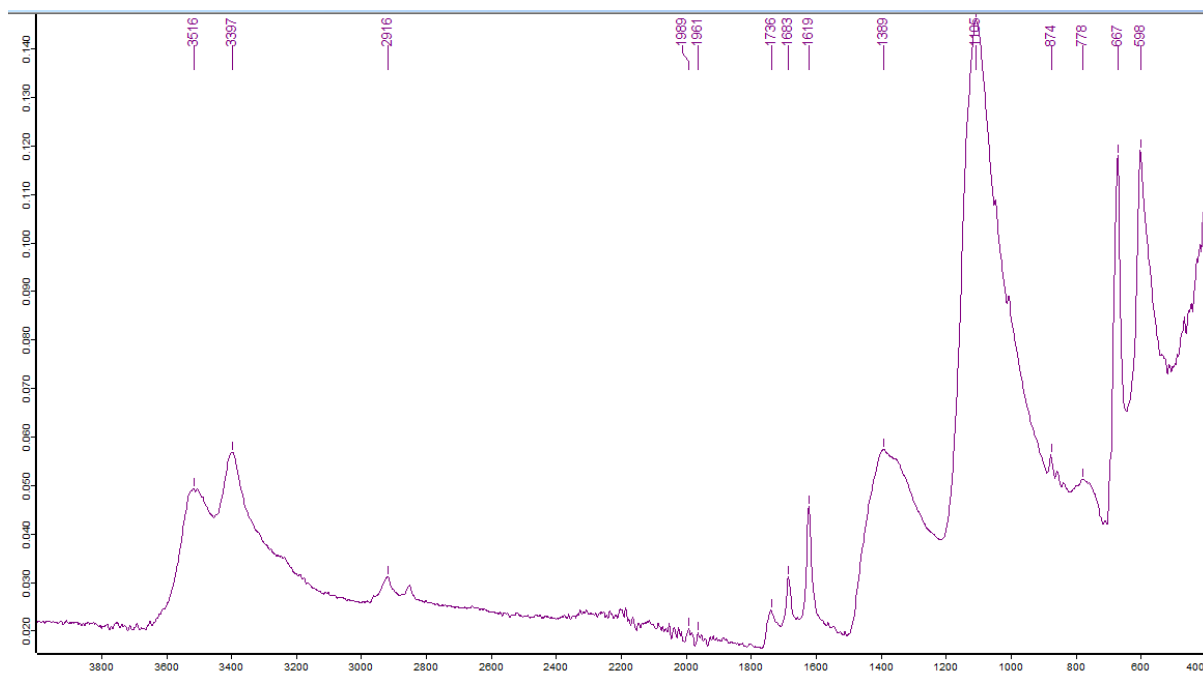


Fig. 64 - FTIR Spectrum. Plaster sample, fragment S9.