# A study of the effect of three commercial cleaning products on shellac-coated mahogany

Ingrid Fitje Apneseth



Master thesis in object conservation Institute for archaeology, conservation and history

UNIVERSITETET I OSLO

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Supervisor: Associate professor Francesco Caruso Institute for archaeology, conservation and history Universitetet i Oslo 2018

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## Abstract

The Royal House of Norway has a large furniture collection from the 19<sup>th</sup> century. Most of this historical furniture is coated with shellac, which was frequently used for such a purpose in the same period. Since these objects are part of the royal collection, they are still in daily use at the different residences. The furniture, therefore, needs a more frequent (and, probably, aggressive) cleaning than what is common for museum objects. For this reason, the effect of three different cleaning products used at the royal collections on shellac-coated mahogany was investigated for the first time.

A thorough study with non-invasive methods (portable FTIR spectroscopy, colorimetry, glossimetry) was undertaken to investigate whether the chemical composition, the gloss and the colour have been altered after the cleaning of varnished wood with the selected cleaning products. The investigations were done on fresh and aged mahogany wood samples coated with shellac. The results of this study show that one of the cleaning products probably causes chemical changes in the shellac.

This study provides information for the ordinary cleaning and subsequent conservation treatments of the above-mentioned collection and aims to create a preliminary non-invasive protocol for investigating varnish-coated wood.

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# **1** Introduction

This chapter focus on the background and research question for the thesis, as well as the way it is structured. The production methods and the chemistry of shellac will then be described, followed by its common deterioration factors.

## 1.1 Background

## 1.1.1 Brief history of the furniture of the Royal Palace

The building of the Royal Palace of Norway began in 1824 and was concluded in 1848 (Kavli, Hjelde, & Hårberg, 1983, pp. 9-16). Its furniture was manufactured throughout the 19<sup>th</sup> century abiding to its different styles (Kavli et al., 1983, pp. 69-78). Since the middle of the 17<sup>th</sup> century, French polishing was a common way to varnish furniture surfaces in Europe, and it was eventually perfected in the 19<sup>th</sup> century (Allen, 1994, pp. 10-11). French polishing is a method by which shellac is applied in multiple layers, followed by polishing to give a highly glossy surface (Rivers & Umney, 2003, p. 19).

## 1.1.2 The reasons for this thesis

The royal collections of Norway have a great amount of varnished furniture. Since most of the furniture is placed in the Royal Palace and other royal residences, the objects are still in everyday use and subjected to the related wear. Because of this, the varnished furniture needs to be cleaned on a regular basis and perhaps in a more aggressive manner, than what is usually the case with museum objects (Schiander, 2017a, personal communication). The royal collections are, therefore, in need of a cleaning routine for their furniture. This should be efficient and should harm neither the varnish layer nor the furniture.

For the aims of this thesis, it was chosen to use wood mock-ups for the cleaning tests, rather than the original furniture. This is in line with the E.C.C.O. professional guidelines and the AIC guidelines for practice, which state that conservators should not perform any unnecessary treatment on cultural heritage and should strive to only use products that – to the best of conservators' knowledge – do not harm the object (E.C.C.O., 2003) (AIC, 1994). As will eventually be shown, in this thesis, mock-ups constituted a realistic replacement to the use of the original furniture for testing purposes. Another important part of E.C.C.O.

guidelines is to strive not to remove material from the object, unless it is indispensable (2003). Keeping this in mind, in this thesis work, only non-invasive methods (with the minor exception of the analysis necessary to determine the thickness of the varnish layer) were employed. Thanks to this approach, the methods used to analyse the mock-ups may be directly employed, to investigate coatings on the furniture from the royal collections, without harming the original materials.

## **1.2 Research question of the thesis**

Conservation methods for treating objects are based on principles of minimal intervention and protection of the original materials (Hanssen-Bauer, 1996, p. 166). Such principles make it necessary to investigate the materials that are considered for the treatment. In fact, materials react differently to various environments and can change over time. Therefore, it is important to investigate the products over time under realistic conditions. By employing nondestructive methods, this thesis investigates the effects that some commercial cleaning products have on a wooden surface treated with aged and unaged shellac.

The main research question is:

How do three commercial cleaning products, affect historical shellac-varnished furniture?

To be able to answer such a question, the following set of **aims** has been set up:

1 – Assess whether the chemical composition, the colour, and the gloss of the shellac coating are altered after cleaning with the commercial products;

2 – Evaluate possible differences in the effects of the cleaning products, between samples with freshly applied shellac and samples that have been artificially aged.

## **1.3 Structure and content**

This thesis is structured as follows:

- In chapter 1, the background, as well as the research question and objectives, for the thesis are described. The production procedure of shellac resin is briefly illustrated. Following this, the general chemistry of shellac is presented with its deterioration factors and mechanisms;
- Chapter 2 gives an overview of the methods and materials employed in the thesis. In particular, the preparation of the shellac-covered samples and the cleaning products that were tested are presented. A detailed account is given for the number of samples, and how the samples differ from each other. Furthermore, the three instrumental methods used for the study are briefly introduced;
- In chapter 3, all the results collected with the methods described in chapter 2 are presented. Schematically, these are divided according to the cleaning products and the treatment;
- Chapter 4 gives a preliminary interpretation of the results and put them into context. This chapter is dedicated to the discussion of the work of this thesis;
- In chapter 5, the conclusion is presented together with an outlook at further avenues of research on this topic.

## 1.4 Shellac

Shellac is an organic resin secreted by the insect *Laccifer Lacca*, as well as by some other similar insects (Horie, 1987, pp. 258-260; Mills & White, 1994, p. 115; Rivers & Umney, 2003, p. 175). The resin is formed and left on the twigs and branches of various host trees, most commonly in India but also in some other south-eastern Asian countries. The shellac is scraped from the trees, washed and purified by sieving the melt (Vandenabeele et al., 2000, p. 269). The resin is then bleached with hypochlorite and the wax separated by saponification (Figure 3). At this point, shellac is shaped into dry sheets. When shellac is applied, the sheets are dissolved in alcohol, creating a varnish that dries by evaporation of the solvent.

#### 1.4.1 Chemistry of shellac

The chemistry of shellac is complex. The chemical composition depends on the host tree where the insects produce the resin (Colombini, Bonaduce, & Gautier, 2003; Tamburini, Dyer, & Bonaduce, 2017, pp. 1-2). Chemically, shellac has two "parts": an ether-insoluble one ("hard") and an ether-soluble one ("soft"). The hard part makes up 70% of the resin, and the soft part makes up 30% (Wang, Ishida, Ohtani, Tsuge, & Nakayama, 1999, p. 1). The hard part mainly consist of oligomers made up by esterification of polyhydroxy carboxylic acid (Mills & White, 1994, pp. 115-117). These acids belong to two different groups. The first one consists of aliphatic compounds related to fatty acids, which are called aleuritic acid and butolic acids. The other group is made of alicyclic compounds of the sesquiterpenes series, mainly, jalaric and laccijalaric acids. These four acids constitute the "skeleton" of shellac.

The hard part is made up of equimolar amounts of aleuritic and sesquiterpene acids (Mills & White, 1994, p. 117). It contains four molecules of each of these, on average, per polymer molecule. The molecule of the hard resin can be seen below (Figure 1), but it must be taken into account that the sequence of linking of the different parts may not be regular or consistent. The molecular weight of this part of the resin is found to be around 2.1 kDa. The soft part of the resin is lighter than the hard one, and is made up of dimers. These are constituted by one molecule of aleuritic acid esterified with a sesquiterpenolic moiety (Mills & White, 1994, p. 117). The soft and hard parts seem to be part of a continuum of oligomers of differential molecular weights, making up the whole shellac molecule.

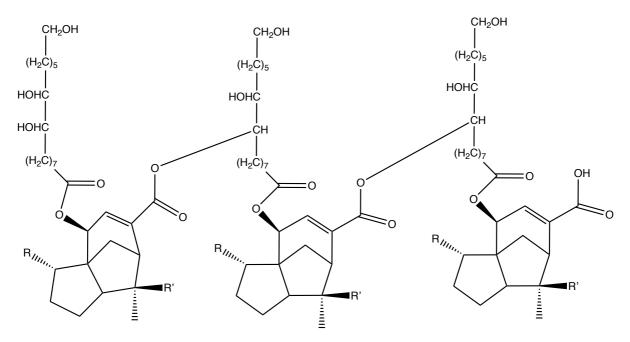


Figure 1: Chemical structure of hard resin of shellac.

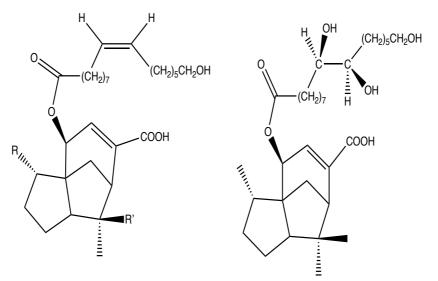
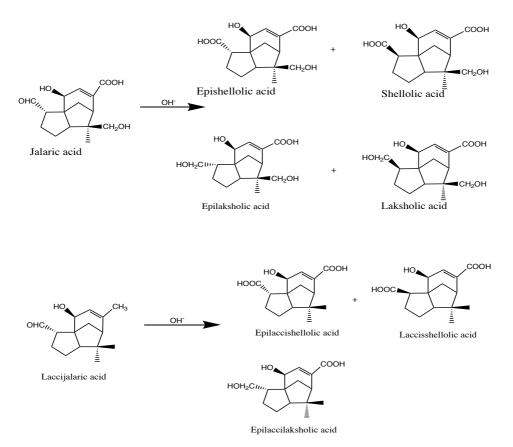


Figure 2 Chemical structure of soft resin compounds of shellac.

An alkaline treatment such as saponification (base hydrolysis of greasy esters), leads to the formation of other compounds, such as in Figure 3.



*Figure 3: Jalaric and laccijalaric acid and the products they make when subjected to alkaline treatments.* 

#### 1.4.2 Deterioration of shellac

According to Rivers and Umney, photochemical deterioration is the main reason for weathering of varnishes (2003, p. 587). Such a phenomenon can cause alterations in the chemical, physical and optical properties of the varnish. Among these alterations, crosslinking, change in solubility, hardness, brittleness, colour and gloss must be mentioned.

Shellac does not constitute an exception to these deterioration processes. As previously mentioned, shellac is chemically formed by different esterification reactions. When applied as a coating it undergoes crosslinking because of self-esterification (Farag, 2010, pp. 14-15). Aldehyde groups therein contained are readily oxidized and converted into carboxyl ones over time (Mills & White, 1994, p. 117). Due to these chemical changes, shellac becomes

less alcohol-soluble. Other changes such as yellowing, cracking, loss of gloss and change in fluorescence can occur due to other chemical alterations (Rivers & Umney, 2003, p. 347). The biggest issue concerning shellac durability is its poor hydrophobicity (Allen, 1994, p. 24). Shellac can absorb water and, subsequently, possibly turn white.

Shellac layers show high mechanical strength and are abrasive-resistant, and it can easily be repaired, if damaged, by applying a new layer on top of the old one (Allen, 1994, p. 24). However, examples of shellac layers on furniture surfaces in good condition can be seen after over a hundred years, thus witnessing its durability (Allen, 1994, p. 24).

## 2 Materials and methods

This chapter focuses on the materials and methods used for preparing and analysing the samples that constitute the object of this investigation. The analysis methods were chosen on the basis of their non-destructivity, their ability to document the samples, to investigate changes in the molecular structure and the surface in addition to evaluating the effect of the cleaning products.

## 2.1 Preparation of samples

African mahogany, Sapele (*Entandrophragma cylindricum*), was used to make the wooden samples (Edlin, 1977, p. 149). The samples were cut with a handsaw into pieces of approximately  $15 \times 7 \times 1.5$  cm<sup>3</sup>, and then sanded with a file to remove the splinters on the edges. The wood varied in colour from deep to light red-brown. Heartwood constitute the darker area of the samples, whereas the lighter part was constituted by sapwood. Since the proportion of heartwood and sapwood in each sample varied considerably, the samples were randomly assigned to the different treatments (see further). The samples were then weighed with an Ohaus Explorer analytical balance (Ohaus corporation, Parsippany, NJ, USA), before being coated with shellac. The length, width and thickness of each sample were measured with a calliper. All of these descriptive measurements can be found in Appendix 1.

The samples were varnished with recto shellac, which is a dark brown coloured shellac, from producer Ernst P Norge AS (Oslo, Norway). Recto shellac was chosen because it has been used at the Royal Palace in recent years to re-varnish surfaces (Schiander, 2017b, personal communication). The shellac was mixed with technical ethanol at a concentration of around 350 g/L. Each wood sample was coated with shellac on one side. The shellac was applied two times with a soft brush. All the samples were left to dry for approximately 24 hours between each application. They were not polished after the application of shellac. Coating was carried out by furniture conservators to obtain homogeneously varnished samples. These conservators work at a conservation workshop called Møbelverkstedet, and have many years of experience with restoring and conserving historical furniture.

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#### 2.1.1 Description of the samples according to their treatment

The wood mock-ups were divided into three different categories according to the tested product and treatment (see further). Each treatment had 9 or 12 samples: three samples for each cleaning product and three control samples (for the categories that involved artificial ageing). Based on these categories the samples were given names that refer to the tested cleaning product and the applied treatment. A scheme of the samples that includes a description of each category can be found in Table 1.

Category	Centurio	Fulgentin	Baolin	Control	Applied treatment
NS	CE.NS1 CE.NS2 CE.NS3	FU.NS1 FU.NS2 FU.NS3	BA.NS1 BA.NS2 BA.NS3		Covered with shellac, and tested for cleaning.
FS	CE.FS1 CE.FS2 CE.FS3	FU.FS1 FU.FS2 FU.FS3	BA.FS1 BA.FS2 BA.FS3	CO.FS1 CO.FS2 CO.FS3	Covered with shellac, tested for cleaning and aged for 240 h.
AS	CE.AS1 CE.AS2 CE.AS3	FU.AS1 FU.AS2 FU.AS3	BA.AS1 BA.AS2 BA.AS3	CO.AS1 CO.AS2 CO.AS3	Covered with shellac, aged for 240 h. and then tested for cleaning.
	9	9	9	6	33 samples

Table 1: Scheme of the wood mock-ups with their names.

#### 2.1.2 Evaluation of the thickness of the shellac layer

The film thickness of the shellac was measured on micrographs acquired with an Olympus BX 60 microscope (Olympus Norge, Asker, Norway), equipped with cross polarised light, at a 200× magnification. An Olympus SC30 microscopy camera was used to take the pictures. The Olympus Stream Essentials Windows program was used to carry out the image analysis. The film thickness was measured on the six control mock-ups. For this purpose, small samples were taken from them. Two micrographs on each sample were acquired, and the thickness was evaluated in three to five points per image (Figure 4). The film thickness of the shellac layer varies across the samples, due to the clustering of pores in the wood (Edlin, 1977, p. 149), but is on average around 10  $\mu$ m on all six samples. The rest of the measurements can be found in Appendix 2.

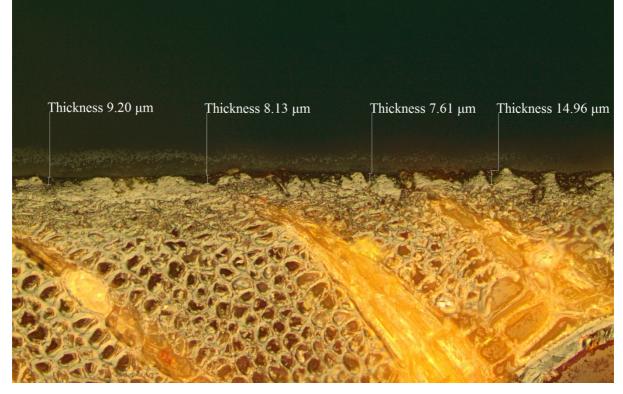


Figure 4: Film thickness measurements from CO.AS3.

## 2.2 Samples ageing

Samples were aged by exposure to UV light and temperature, which are deteriorating factors for shellac. The samples were aged in a QUV aging chamber, QUV/spray (Q-lab, Westlake, OH, USA). The duration of the treatment in the ageing chamber was chosen with the help of the literature, it was chosen to age the samples for 240 hours (Çakicier, Korkut, Korkut, Kurtoğlu, & Sönmez, 2011; Sarkar & Shrivastava, 2000). The UV light was turned on for the whole period and the intensity was set to 0.3, whereas the temperature in the chamber was set to 45 °C. To homogenize the treatment as much as possible, the samples were moved once in the chamber during the ageing process, because the intensity of the UV light slightly varied in the different areas of the chamber. Only a part of each sample (an area of  $6.3 \times 9.5$  cm<sup>2</sup>) was exposed to UV light during the ageing treatment, while the rest of the sample was covered by the metal stand.

## 2.3 Cleaning products

Three commercial cleaning products were tested. This choice was made on the basis of the commercial availability of the products, their chemical composition and their use in furniture care. These commercial cleaning products were sold as ready to use. In the next paragraphs, a rapid description of each product is given.

## 2.3.1 Centurio Möbelpolish

Centurio furniture polish is produced by Ernst P. Norge AS (Oslo Norway). According to the producer, the constituents of Centurio are oil, water and a fine-grained abrasive in an unknown proportion<sup>1</sup> (A. B. Ernst P). The product looks opaque and light pink in colour. Centurio was chosen because it is frequently in use at the Royal Palace (Schiander & Skipperud, 2017, personal communication). Centurio is also regularly used by other conservators for the cleaning of furniture surfaces (Bjørk, 2017, personal communication) (Marano, 2014, p. 32).

## 2.3.2 Baolin Möbelpolish

Baolin Möbelpolish is a cleaning product, also by Ernst P. Norge AS. The composition can be found in Table 2. Baolin is transparent and light yellow in colour. Baolin was chosen because it is sometimes used by the furniture carpenters of the Royal Palace (Schiander & Skipperud, 2017, personal communication). It was also chosen because it has a different chemical nature compared to the Centurio product (N. A. Ernst P, 2015).

Constituents	Mass content
Hydrocarbons C <sub>11</sub> -C <sub>12</sub> , Isoalkanes,	50-100%
<2%aromatic	
Mineraloils solvent dewaxed, light	15-30%

Table 2: Composition of Baolin Möbelpolish, as reported in the accompanying documentation (N. A. Ernst P, 2015).

<sup>&</sup>lt;sup>1</sup> There is no information about the contents of Centurio or at which quantity it is present.

## 2.3.3 Fulgentin Polermedel 100

Fulgentin is produced by MAPA Produkter (Limhamn, Sweden). The composition of Fulgentin is shown in Table 3. The cleaning product is opaque and white coloured. Fulgentin was chosen because of its high commercial availability. It is however not as familiar to furniture conservators as the other cleaning products. The composition of Fulgentin has similarities with Centurio, as they are both water-containing products, but there is no mention of any abrasive.

Table 3: Composition of Fulgentin Polermedel 100, as reported in the accompanying documentation (MAPA).

Constituents	Mass content
Aliphatic hydrocarbons C <sub>10</sub> -C <sub>13</sub>	10-15%
Mineral oils	30-40%
Water	50-60%
Dispersants	< 0.2%

#### 2.3.4 Application of cleaning products

The three cleaning products were applied in the same way. They were applied on the samples with a new, clean and dry terrycloth (Leon klut, KID interiør, Gullaug, Norway). This type of terrycloth was suggested by a furniture conservator on the basis of her work experience (Hagen, 2017, personal communication). The cleaning products were applied with straight movements, in the same direction as the fibres of the wood. The cloth was dipped in the product and applied evenly on the sample. The cloth was dipped in the cleaning product a second time and applied on the sample, so that some excess of product was still on the sample. The product was left to rest for one minute. Afterwards the residues were wiped off with another clean, dry terrycloth. This application method was chosen on the basis of the instructions accompanying the products. After the application of the cleaning products, the samples were left for 10 days to allow the volatile residues to evaporate.

## 2.4 Analytical Methods

In Figure 5, an overview of the treatments and the measurements carried out in this thesis work can be found.

Temporal sequence						
		NS samples				
Measurements		Cleaning	Ν	leasurements		
		FS samples				
Measurements	Cleani	ng	Ageing	Measurements		
		AS samples				
Measurements	Ageing	Measurements	Cleaning	Measurements <sup>*</sup>		

Figure 5: Sequence of the treatments (ageing and cleaning) and measurements carried out on the samples. \* Colorimetry measurements were done 7 days after cleaning, while the FTIR measurements were done 10 days after cleaning.

## 2.4.1 FTIR

Infrared (IR) spectroscopy is an established analytical method used for characterizing inorganic and organic compounds (Derrick, Stulik, & Landry, 1999; Kozaris et al., 2013, p. 66; Stuart, 2007, pp. 110-118). This technique records the vibrations within a molecule, when this absorbs the IR radiation between 700 nm (frequency 430 THz, wavenumber ca. 14 300 cm<sup>-1</sup>) and 1 mm (frequency 300 GHz, wavenumber 10 cm<sup>-1</sup>). Vibrations can come in various forms (depending on the bond forces and the atomic masses of the atoms at stake) and can involve variations of both bond lengths and angles (Stuart, 2007, p. 110). Movements like these can change the dipole moment of a molecule. For a molecule to be IR-active, the dipole moment needs to change. An IR spectrum can, therefore, be considered a fingerprint of a certain substance and IR spectroscopy can be used to evaluate the ageing (when this results in a chemical change) of different substances (Derrick et al., 1999, p. 133).

In this work, FTIR was chosen to evaluate the ageing of shellac and to investigate whether any other changes have occurred after the treatment with the cleaning products. The FTIR measurements were performed with a 4300 handheld FTIR by Agilent technologies (Santa Clara, CA, USA) (Figure 6). The samples were analysed in external reflection mode. External reflectance (ER) were used because of the glossy surface of the samples (Chalmers, Everall, & Ellison, 1996; Derrick et al., 1999, pp. 59-62; Stuart, 2015). Spectra were obtained between 4000 and 650 cm<sup>-1</sup> by accumulation of 16 scans at a spectral resolution of 4 cm<sup>-1</sup>. A background scan was collected every 60 minutes during all test periods. Spectra were acquired on both sides of the samples and, on each side, spectra on four different points were collected. Spectra were not baseline-corrected.



Figure 6: The handheld FTIR.

## 2.4.2 Colorimetry

Colorimetry is a technique to measure the colours of objects (Berns, 2016; Johnston - Feller, 2002; Koenderink, 2010). This method is often used for documentation and is a way to determine if colour changes have occurred (Berns, 2016, p. 17). For this analysis, a handheld Konica Minolta Chroma Meter CR-400 (Konica Minolta Sensing Europe B.V., Nieuwegein, the Netherlands) with an 8-mm measuring aperture was used (Figure 7). Measurements in both specular component excluded (SCE) and specular component included (SCI) are reported in the CIELAB colour space.

In this work, colorimetric measurements were used to investigate whether any colour change had occurred between any of the stages undergone by the samples. 20 repeated measurements were carried out on each side to obtain statistically significant colour values.

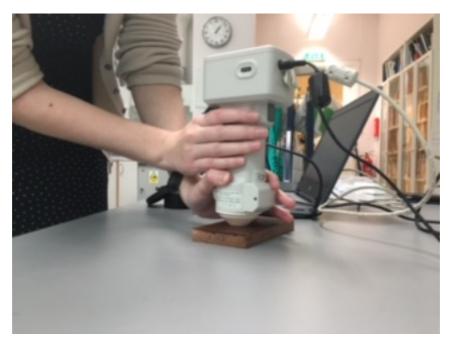


Figure 7: The spectrometer.

#### Explanation of the different colour values

#### SCE and SCI

When talking about colour change, it is important to know how the colour measurements work and what the different categories of values mean. The values have first been divided into SCI and SCE values. To understand the variations in SCE and SCI, it is important to know the difference between specular and diffused reflection. Specular reflection is the phenomenon that occurs when light is reflected in the same but opposite angle from where the light originated (Konica-Minolta, 2003, p. 32). Diffused reflection occurs when the light is scattered in many different directions. The sum of these two reflections is called the total reflectance. Therefore, surfaces with high reflectance, such as highly glossy surfaces, have a high specular reflection, whereas the opposite is the case for low gloss surfaces.

Specular component excluded (SCE) is the way by which the spectrometer measures the colour without the specular reflectance while specular component included (SCI) also includes the specular reflectance in the measurements (Konica-Minolta, 2003, p. 33). This means that in SCE the diffuse reflectance is measured, which is more in line with the colour that is visible for an observer. Since SCI also measures the specular reflectance, it is more

representative for the total appearance of the colour. As these two modes measure different variations in colour, it was important to consider both these variations. This has been done in this thesis, as the spectrometer used could measure SCE and SCI at the same time. In this thesis the focus will be put on the SCE results for the samples, this is done because the samples have a glossy surface, and it is more descriptive to exclude the specular reflectance when considering the colour change.

#### CIELAB

The way of measuring colours in  $L^*$ ,  $a^*$  and  $b^*$  was defined in 1976 by CIE, and it is called CIELAB system, it is the method used in this thesis (Konica-Minolta, 2003, p. 11).  $L^*$  represents lightness (the different variations between black and white), whereas  $a^*$  (the variation between red and green) and  $b^*$  (the variation between yellow and blue) represents chromaticity coordinates. Changes in values of either of these variables mean that there is a shift towards one of the mentioned colours.

#### 2.4.3 Gloss measurements

Visual analysis was undertaken to characterise the gloss of the surfaces of the wood samples treated with shellac. The gloss was evaluated with the method described by Hoadley (1980, p. 180 &189). A sheet of paper with vertical, horizontal and diagonal lines was held at a 90° angle on the surface. Based on the distortion of the pattern on the surface of the shellac, the gloss was evaluated as "very good", "good" and "poor". All the samples were photographed during gloss measurements, to be able to compare the samples before and after treatment.

## **3 Results**

In this chapter all the results of the artificial ageing and the cleaning tests will be presented. The results of the artificial ageing are described first followed by the cleaning tests. The cleaning tests are presented individually for each cleaning product.

## 3.1 Ageing treatments

## 3.1.1 FTIR

The spectrum of sample CO.FS1 (treated with unaged shellac) is shown in Figure 8. This shows peaks at 2950 and 2878 cm<sup>-1</sup>, which refer to C-H stretches. A small peak is present at 2360 cm<sup>-1</sup>. There is a strong peak at 1741 cm<sup>-1</sup>, which indicates a C=O stretch from either an ester or an acid. The peak at 1470 cm<sup>-1</sup> can refer to a C-H bending vibration. Furthermore, there are peaks present at 1280, 1173 and 1088 cm<sup>-1</sup> that indicate C-O stretches from either esters, acids or alcohols. The small peak that is visible at 950 cm<sup>-1</sup> can indicate a C-H stretch. Keeping into account the spectral changes induced by the ER mode, these are all characteristic peaks for shellac (Derrick et al., 1999, p. 107).

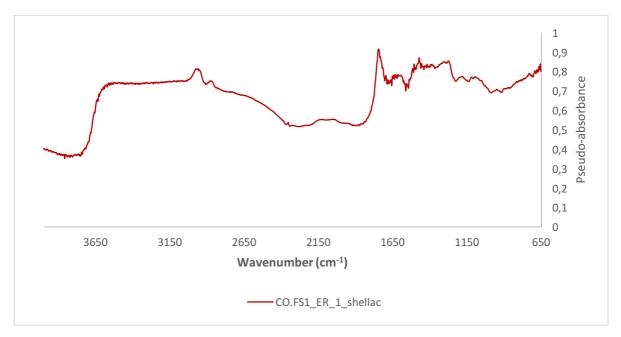


Figure 8: ER FTIR spectrum from sample CO.FS1.

Comparing the spectrum of a sample of wood covered with unaged shellac with the spectrum of a sample with aged shellac (Figure 9), we can notice that the main difference is the peak around 2360 cm<sup>-1</sup>. The intensity of this peak has slightly increased on the aged shellac samples (Figure 10). This is present in all the samples that underwent the ageing process. Another difference can be found for the peak around 1750 cm<sup>-1</sup>. This peak has slightly increased in intensity in all but three of the measurements taken on the aged samples (Figure 11). Two of the measurements of the aged shellac samples, have peaks with lower intensity than before ageing, whereas one has a similar intensity. The rest of the spectra can be found in Appendix 3.

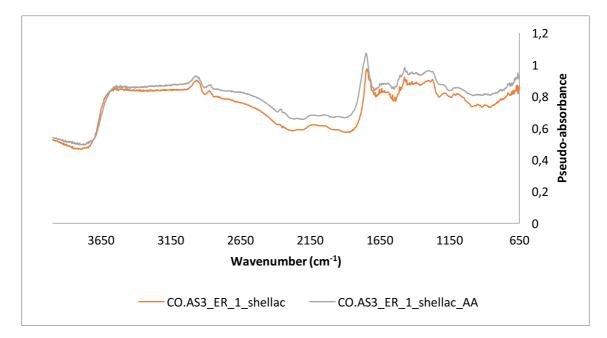
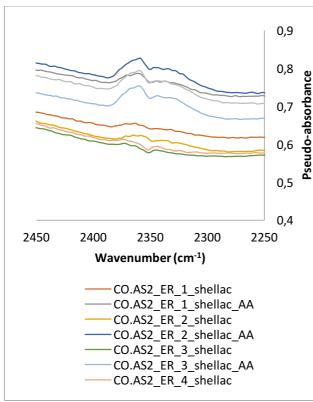
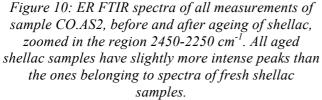


Figure 9: ER FTIR spectra of sample CO.AS3 before and after ageing.





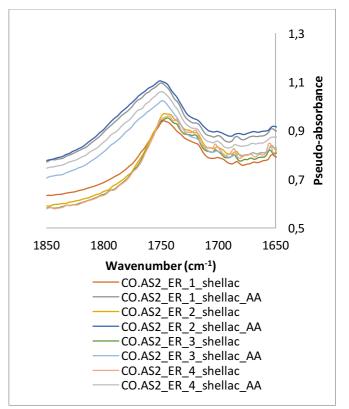


Figure 11: ER FTIR spectra of all measurements of sample CO.AS2, before and after ageing, zoomed in the region 1850-1650 cm<sup>-1</sup>.

#### 3.1.2 Colorimetry

All the colorimetric data acquired for the aged samples, underwent the IOS-recommended Grubbs' test for the exclusion of outliers with P = 0.05 (Miller, 2010, pp. 49-50). To evaluate whether there had been a significant change from before and after cleaning, t values were calculated for  $L^*$ ,  $a^*$  and  $b^*$  (Miller, 2010, pp. 38-40). If each stage had more than two values above t critical levels the sample were deemed to have a significant change in colour<sup>2</sup>. These calculations are the same for all the colorimetric data acquired for the cleaning products described below. All the results, including the SCI ones, can be found in Appendix 4.

<sup>&</sup>lt;sup>2</sup> t critical is 2.09, when having 20 measured points, and 2.10 when having 18 measured points. (Miller, 2010, p. 266)

When reviewing these results, two out of 12 of these samples, showed significant changes in their SCE colour values. These are samples FU.AS2 and BA.AS3. For both of them, their  $L^*$  and  $a^*$  values changed. In both cases the  $L^*$  and  $a^*$  values increased, meaning that they became lighter and redder in colour. The results in SCI mode of sample CO.AS3 also showed a significant change, with an increase in  $L^*$  and  $b^*$  values.

#### 3.1.3 Gloss measurements

The gloss measurements of all the samples that went through the weathering chamber had some differences between the aged part of the sample and the area covered by the metal stand. These were perceived as a slight change in gloss, and this change was more evident in some samples than others. It was however difficult to say anything about the degree in which the gloss had been altered. Below is an example (Figure 12). The rest of the gloss measurements can be found in Appendix 5.

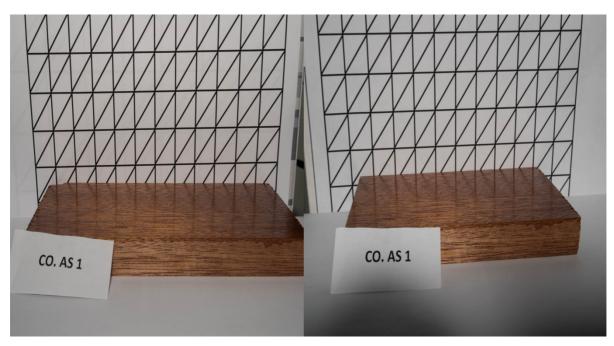


Figure 12: Gloss measurements of sample CO.AS1. Before ageing to the left, and after ageing to the right.

### 3.2 Cleaning tests with Baolin

#### 3.2.1 FTIR

When reviewing all the FTIR spectra of the samples cleaned with Baolin, these – before and after cleaning – all have the same characteristic peaks of shellac. There are no changes in the ER FTIR spectra after the cleaning product was applied, as can be seen for sample BA.AS3 in Figure 13. The rest of the results can be found in Appendix 3.

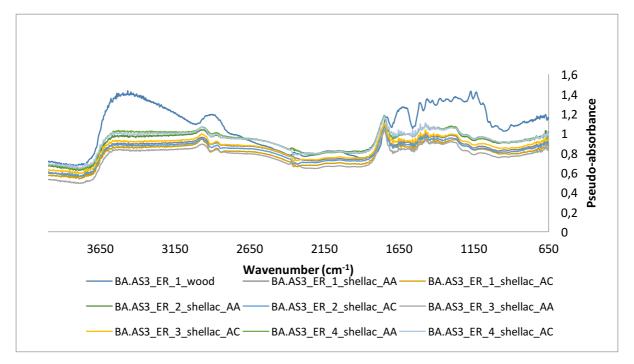


Figure 13: ER FTIR spectra of sample BA.AS3 before and after cleaning with Baolin.

#### **3.2.2** Colorimetry

When reviewing these results, two out of nine of the samples cleaned with Baolin showed significant changes in their colour values. Samples BA.NS1 and BA.AS3 were the ones where the colour changed in the SCE values. Both of these samples had a significant change in the  $L^*$  values and both of these moved towards a darker colour. BA.NS1 also had a change in  $b^*$  where the colour shifted towards yellow and BA.AS3 had a change in  $a^*$  where the colour shifted towards green. There were also some changes in the SCI values these changes involved three of the samples cleaned with Baolin BA.FS3, BA.NS1 and BA.AS3. Figure 14 shows an example of significant changes in the SCE values of BA.NS1. The rest of the colorimetry results can be found in Appendix 4.

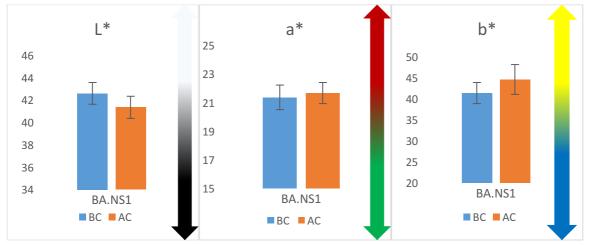


Figure 14: Colorimetry results for BA.NS1 before and after cleaning with Baolin. These histograms are based on a total of 40 measurements.

#### 3.2.3 Gloss measurements

For the samples cleaned with Baolin, there were no apparent differences in gloss, from before and after the cleaning. All the pictures of the gloss measurements can be found in Appendix 5.

# 3.3 Cleaning tests with Centurio

#### 3.3.1 FTIR

All FTIR spectra of the samples treated with Centurio show changes after the cleaning. The spectra show changes in the fingerprint region (Figure 15). In the spectra of the samples before cleaning, a peak at 1280 cm<sup>-1</sup> and two broad peaks at 1173 and 1088 cm<sup>-1</sup> are present. After cleaning, two narrow peaks with higher intensity are present at 1050 and 1010 cm<sup>-1</sup> (Figure 16). A peak is also present at 815 cm<sup>-1</sup>, and a more pronounced peak can be seen around 950 cm<sup>-1</sup>. The rest of the spectra can be found in Appendix 3.

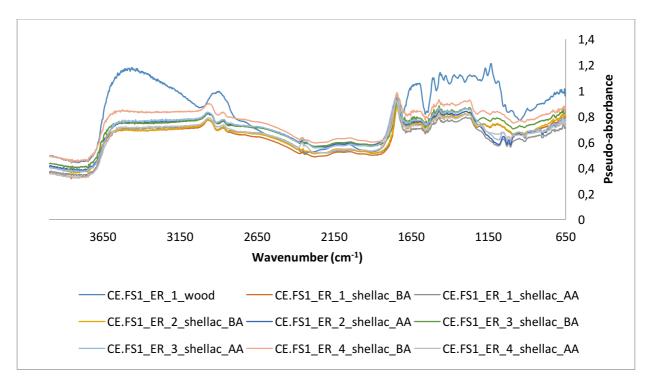
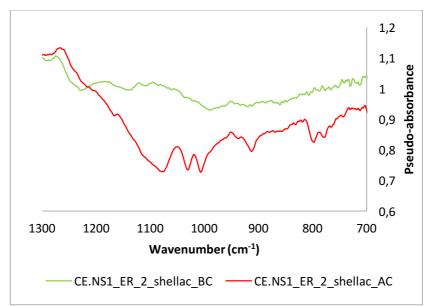


Figure 15: ER FTIR spectra of all measurements done for sample CE.FS1.



*Figure 16: ER FTIR spectra of sample CE.NS1. Zoomed in on area 1300-700 cm<sup>-1</sup>. Alterations in the spectra of cleaned shellac are visible.* 

#### 3.3.2 Colorimetry

When reviewing these results, eight out of nine samples cleaned with Centurio showed significant changes in their colour values. Sample CE.AS1 did not have any significant colour changes after cleaning. Because of the number of samples and changes, the three colour variables will be described separately.

#### L\* values

Concerning the lightness changes, in the samples cleaned with Centurio, there were six samples that had significant changes in the  $L^*$  values. All of them had a change toward a lighter colour. Samples CE.NS3 and CE.AS2 did not have any significant changes in  $L^*$  values.

#### a\* values

For what concerns the  $a^*$  values, there were six samples that had a significant change, similar to the  $L^*$  values. These samples were all following the same trend, moving towards a greener colour. CE.NS2 and CE.FS3 did not have any changes in the  $a^*$  values that can be deemed significant.

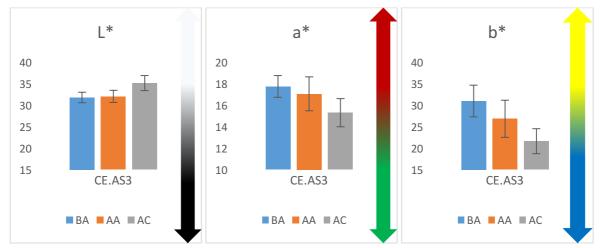
#### b\* values

When looking at the results for the  $b^*$  values, all eight samples had a significant change. All of them shift towards a bluer colour.

#### **SCI values**

When reviewing the SCI results, only sample CE.NS2 had any significant change. These changes were made to the  $a^*$  and  $b^*$  values. These changes do however show the complete opposite of the trends seen in the SCE values, as the  $a^*$  shift towards red, and the  $b^*$  values moves towards yellow. But as it is only one of the samples that has these changes it is impossible to say whether this change is consistent or not.

An example of the SCE values can be found in Figure 17, while the rest of the results can be found in Appendix 4.



*Figure 17: Colorimetry results for CE.AS3, before, after artificial ageing and after cleaning with Centurio. These histograms are based on a total of 57 measurements.* 

#### 3.3.3 Gloss measurements

For the samples cleaned with Centurio, there appear to be a difference within the samples. For the NS and FS samples there do not appear to be a difference from before and after cleaning. But in the aged shellac samples, that were then cleaned with Centurio, there seem to be a loss of gloss. All the pictures of the gloss measurements can be found in Appendix 5.

# 3.4 Cleaning tests with Fulgentin

#### 3.4.1 FTIR

The FTIR results of the shellac samples cleaned with Fulgentin are all showing consistent results throughout the treatment, as can be seen in Figure 18. There are no substantial differences in the spectra before and after treatment. The only spectral area that shows some slight differences is the same as the one of the aged samples, around 2360 cm<sup>-1</sup>. The remaining spectra can be found in Appendix 3.

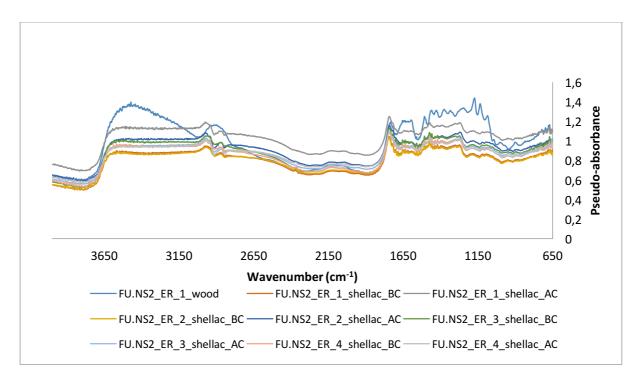


Figure 18: ER FTIR measurements of sample FU.NS2, before and after cleaning with Fulgentin.

#### 3.4.2 Colorimetry

When reviewing these results, three out of nine of the samples cleaned with Fulgentin showed significant changes. FU.FS1 and FU.FS2 were the ones that had colour change for the samples cleaned with Fulgentin. Both of them had a change in their  $b^*$  values, where they shifted towards a bluer colour. For FU.FS1,  $a^*$  moved towards red, and for FU.FS2, the  $L^*$  became higher (lighter). Sample FU.NS1 showed significant changes in SCI values. An example of FU.FS1 can be seen in Figure 19. The rest of the colorimetri results can be found in Appendix 4.

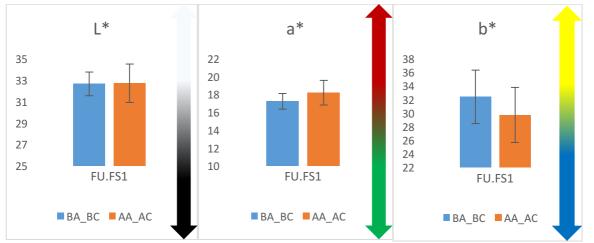


Figure 19: Colorimetry results for FU.FS1, before and after artificial ageing and cleaning. These are based on a total of 40 measurements.

### 3.4.3 Gloss measurements

For the samples cleaned with Fulgentin, there were no apparent differences in gloss, from before and after the cleaning. All the pictures of the gloss measurements can be found in Appendix 5.

# **4** Discussion

This chapter covers the interpretation of the results presented in chapter 3. The discussion of the results is divided according to the different analytical methods employed. Other issues relating to the work undertaken are also discussed.

### **4.1 FTIR**

#### 4.1.1 Aged shellac samples

When comparing the FTIR spectrum of a sample covered with unaged shellac to the spectrum of the same sample covered with the aged resin, there are certain changes in band width and intensity that are expected to be present: namely a broadening of the peak around  $3400 \text{ cm}^{-1}$ , an intensification of the peak at  $1730 \text{ cm}^{-1}$  as well as a change in the fingerprint region concerning the C-O bands ( $1240-1040 \text{ cm}^{-1}$ ) (Striova et al., 2015, p. 95). Derrick et al. also state that the greatest change in a spectrum after ageing concerns the intensity of the carbonyl band (1999, p. 105). These changes relate to the esterification that shellac undergoes with ageing. An increase in intensity of the carbonyl peak was evident in the aged shellac samples (Figure 11). Another pronounced change in the aged samples pertains the doublet around 2360 cm<sup>-1</sup>. This doublet has increased in intensity in most of the samples investigated after ageing. However, this doublet is most likely the result of atmospheric CO<sub>2</sub> presence (Pavia, Lampman, Kriz, & Vyvyan, 2014, pp. 86-87). There are rarely other functional groups that absorbs around 2350 cm<sup>-1</sup>, and CO<sub>2</sub> has a characteristic doublet that can be easily recognised.

Regarding the other change in the spectra relating to the C=O stretch (1750 cm<sup>-1</sup>), this indicates that esterification has occurred when the samples underwent ageing. Following this, there should be a change around 3400 cm<sup>-1</sup>, as the increase of OH groups is expected together with the increase of esterification (Striova et al., 2015, p. 95). There is however no clear peak referring to OH stretching in any of the recorded spectra. There is a slight signal between 3600 and 3500 cm<sup>-1</sup> which could refer to such a bond. Striova et al. states that this peak should not be found any higher than 3410 cm<sup>-1</sup> (2015, p. 95). However, Scheinmann gives a range for this peak from 3200 to 3550 cm<sup>-1</sup> (1970). If this is the case, the weak signal can be the result of the stretching of OH bands. Not getting a clear signal of the OH stretching is however uncommon

for infrared spectra of shellac. The OH functional group is a very important part of the shellac molecule, as they refer to acids in the structure. This can also be influenced by the instrument in which the spectrum is taken. These spectra have all been measured with a handheld FTIR and there is no real database of spectra to compare with. Therefore, there can be alterations in what can be expected in the spectra, which results in some of the peaks that are expected to be there are not present. In addition to this the literature referred to in this part, has not used ER FTIR in their work, and it is important to mention that when using ER FTIR the spectral regions can shift. So some of these changes might be a result of the ER geometry.

#### 4.1.2 Samples cleaned with Baolin and Fulgentin

Looking at the FTIR measurements for all the samples cleaned with Baolin and Fulgentin, these results show the same trends. The spectra of the samples before and after applying the cleaning product show no significant changes. The only significant change is around 2360 cm<sup>-1</sup>, and as explained in the previous part this probably refers to the presence of  $CO_2$ , and does not have any impact on the shellac coating. On the basis of the results from the tested samples, this is a good indicator that these cleaning products have not affected the shellac in a way that it detectable with ER FTIR. This might indicate that these two cleaning products do not contain substances that have altered the chemistry of the shellac coating. There do not seem to be any difference between aged samples of shellac or freshly applied shellac, in regards to the way the samples have reacted to the cleaning products.

Even though these results indicate that the Baolin and Fulgentin cleaning products do not cause any chemical alterations on the coating, it does not mean they are suitable to use. These results indicate that in the timeline tested, which is a quite limited one, and with the technique employed for the study, these products did not seem to react with shellac. It is important to consider that these products can cause changes to the shellac in a long-term perspective rather than the allotted time for this research. Due to different chemical components in the cleaning products, they can react differently and due to different factors, and also at shorter or longer intervals. Therefore, a longer study should be carried out to investigate such long-term effects.

#### 4.1.3 Samples cleaned with Centurio

Unlike the results from the cleaning tests involving Baolin and Fulgentin, the results from the cleaning with Centurio show different alterations in the FTIR spectra. This indicates that there might have been changes in the chemical composition of the shellac, due to the absorbance at different wavenumbers. As these changes are consistent throughout the samples cleaned with Centurio, and that they are not present on any of the other samples cleaned with Baolin and Fulgentin, this implies that the treatment with Centurio is the cause for these changes.

Since the changes in the spectra of the cleaned samples are in the fingerprint region, the focus will be on this part of the spectra. Before the cleaning with Centurio, a peak at 1280 cm<sup>-1</sup> was observed, there were also two broader peaks at 1170 and 1088 cm<sup>-1</sup> (Figure 15 and Figure 16). These peaks are stretching absorptions of C-O groups from acids, esters and alcohols (Derrick et al., 1999, p. 107). After cleaning with Centurio, the peak around 1280 cm<sup>-1</sup> is still present, but the spectra show distorted bands (derivative-like and *reststrahlen* ones probably) (Miliani, Rosi, Daveri, & Brunetti, 2012) followed by weak peaks at 1050 and 1010 cm<sup>-1</sup>. Another change that has occurred after cleaning is the appearance of a peak at 815 cm<sup>-1</sup>, and there was an increase of the intensity of the peak at 950 cm<sup>-1</sup>. The peaks around 1050 and 1010 could be a shift in the spectrum so that these still are absorptions of C-O in esters, acids or alcohols. It is most likely in alcohols, since the values are so low, in esters or acids the values would normally be higher(Sigma-Aldrich, 2018). These C-O absorptions are indicators of C-O stretches. As these peaks have shortened and increased its intensity this can be an indication that there is a larger presence of these functional groups, because of alterations in the shellac molecule. The peak at 950 can indicate a C-H bending, and the fact that this peak has increased its intensity, can indicate an increase in C-H bond (Pavia et al., 2014, pp. 86-87). The absorptions around 815 cm<sup>-1</sup> are also most likely signals for C-H bending.

To identify more closely what happens during cleaning would require more analysis, and based on the results from this work it is not possible to determine what happens to the shellac molecule.

#### 4.1.4 Possible residues of cleaning products

One of the possibilities that was investigated after reaching these results was whether or not residues of the cleaning product on the surface could possibly still be present after the cleaning procedures and – as a consequence – alter the recorded spectra. There are some substances that can disturb FTIR spectra and mask other absorptions of the substance, one such substance is water (Minnes et al., 2017). Because of this, it was decided to investigate whether there could be any residues of cleaning products on the surface of the samples varnished with shellac. Considering that all the cleaning products were applied in the same manner, this means that the possibilities of residues should not only be concerned for the Centurio-treated samples. There is however a difference in the texture of the three tested cleaning products. This may influence the probability of leaving residues, even though all samples were wiped with a dry cloth.

In view of such an occurrence, the cleaning products alone were analysed by FTIR and these measurements can be found in Appendix 3. In this way, it was possible to obtain spectra from the cleaning products themselves, giving a chance to compare the spectrum of the pure Centurio with the mock-ups cleaned with it, to see if the recorded changes of the spectra of the cleaned samples are similar to the features present in the spectrum of Centurio. However, FTIR is a technique that is very sensitive to water, and it was therefore necessary to apply the cleaning products on glass slides and leave them to dry for 2-3 weeks. The glass slides with Centurio and Fulgentin were not completely dried and remained sticky after this period. This made the process of acquiring spectra more difficult. Some spectra Centurio were however recorded in ER mode. To prevent the sticky semi-dried cleaning products to attach to the instrument, there was no contact between the samples and the FTIR instrument. This created an uncertainty if the measured spectra were really representative for Centurio. Therefore, it was decided to measure Fulgentin and Centurio in ATR mode, because, with such an interface, the cleaning product could be easily wiped off. Baolin was also measured in ATR. As the peaks are somewhat different in the ATR spectra than in the ER spectra it can be difficult to compare these to each other.

When looking at the ER FTIR spectrum of pure Centurio, this has a number of peaks (Figure 20). There is a strong peak at 2960 cm<sup>-1</sup>, followed by a smaller peak at 2860 cm<sup>-1</sup> (both refer to C-H stretching) (Pavia et al., 2014, pp. 86-87). It also has two peaks closer to the

fingerprint region at 1477 and 1384 cm<sup>-1</sup> (C-H bending). It has a doublet centered at around 1120 cm<sup>-1</sup>(C-O stretching), where the first peak is much more intense than the second. There is also a broad peak at 944 cm<sup>-1</sup>(C-H bending). When comparing this spectrum to one of a sample treated with shellac and eventually cleaned (Figure 21), these are different. However, when considering the fingerprint area in particular it has similar qualities, with more pronounced peaks in this region, something the shellac did not have before the cleaning. This can indicate that the changes in the spectra of the samples covered with shellac, after cleaning with Centurio can be caused by its residues on the surface. It is however difficult to conclude anything without further testing.

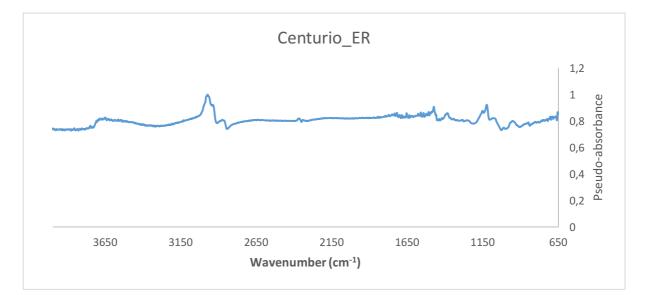


Figure 20: ER FTIR spectrum of Centurio.

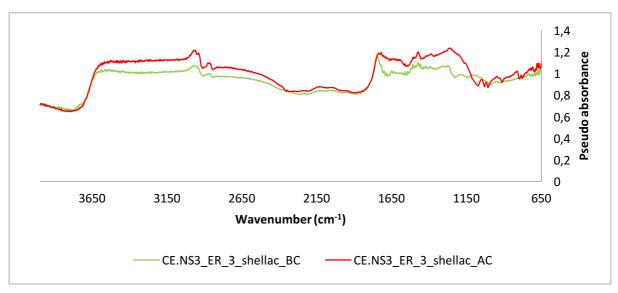


Figure 21: ER FTIR spectra of CE.NS3.

### 4.2 Colorimetry

#### 4.2.1 Colour change

It is important to consider what factors can make colours change, and also in regards to shellac, what these changes might often lead to. Shellac is a substance that is prone to changes in the optical properties. These changes often include yellowing and loss of transparency (Rivers & Umney, 2003, p. 587). Shellac is also at risk of darkening over time (Puică & Oancea, 2008, p. 64). Light is a source that is often an activation energy for the chemical reactions that often cause deterioration (Rivers & Umney, 2003, p. 348). For coatings it can absorb the energy and this again cause bond breaking. Such bond breaking can cause alterations to the electron arrangement and thus preventing electron excitement. And this electron excitement is the reason for the colours observed on the surface, this process can therefore, result in permanent fading or colour change. Therefore, UV radiation is a primary factor that causes colours to change, especially fading. This is the reason why UV radiation was included in the ageing process of the samples: to see whether it had any effect on the shellac coating and how big the extent of such an effect was.

#### 4.2.2 Aged samples

As described, above the expected changes for shellac surfaces when it ages is yellowing and a darkening of the surface. When considering the two samples that had a significant change in colour after ageing they became lighter and redder in colour. There was no shift in the *b*\* where the yellow colour coordinates are. These changes are different from what could be expected when shellac is aged, but as the change is only present in two out of 12 samples, it does not constitute a majority, and it is difficult to draw conclusions from the data. Sample CO.AS3 showed significant changes in the SCI values, and here the colour shifted towards a lighter yellow colour. This is more in line with what could be expected compared to the SCE data, but again this is only one sample and it is not enough data to reach any conclusions.

#### 4.2.3 Samples cleaned with Baolin and Fulgentin

The results showed that only two samples treated with Baolin and two with Fulgentin, had a significant colour change in SCE mode. For the Baolin cleaned samples the common change in both samples where that the colour became darker. This can indicate that this is colour change that occur when cleaning with Baolin. For the two samples cleaned with Fulgentin the common change was a shift towards a bluer colour. This might suggest that an increase in the blue colour, occur when cleaning with Fulgentin. However, for both cleaning products only two out of nine samples showed these changes, and it is not enough samples to reach any conclusions on. For the Fulgentin samples both the significant changes occurred in the fresh shellac (FS) samples, this can be an indication that fresh shellac might be more vulnerable to changes to the colour than the other categories.

#### 4.2.4 Samples cleaned with Centurio

As described in the results, eight out of nine samples cleaned with Centurio had a significant colour change. Six samples became lighter, six samples became greener and all eight samples shifted towards a bluer colour. This constitutes a majority of the samples, which means that this is an observed systematic colour change that can be attributed to the treatment with Centurio. As there were only one sample that did not have any significant change, all categories of samples had changes to their colour. This show that there is no ageing stage of the shellac that reacts differently to the treatment with shellac, as all of them are affected by changes. These results show that the changes in which the colour changes are consistent throughout all the samples cleaned with Centurio. The fact that all the samples follow the same trend is in line with the results obtained from the FTIR measurements.

#### 4.2.5 Visibility of colour change

When looking at the results from the colorimetry, there are some, especially within the Centurio cleaned samples, that have a significant colour change. But even though there is a substantial colour change, it is not always of great numerical value. Therefore, it is important to consider whether the colour change is visible to the naked eye after cleaning. The colour change for most of the samples is not visible to the naked eye, when consulting the pictures in Appendix 6. There is however a slight change in some of the samples cleaned with

Centurio, where some seem less saturated after cleaning than before. When looking at the AS samples there is a difference between the aged and cleaned part of the samples and the part that was covered during the ageing process. This can be the result of a colour change or a loss of gloss, that gives a different perception of the samples. It is most likely that this is a change in gloss. This theory is strengthened by the colour measurements, where only three of them has substantial changes after ageing. This should indicate that the colour change is of such minor values that it should not be visible to the naked eye. When looking at the aged shellac samples for Centurio this divide between aged and unaged shellac seem to be more visible after cleaning.

It must also be mentioned that the pictures of the samples were taken with different angled lighting. This was because it was not done a detailed account of how the lighting was during the first photo session. This has affected the pictures in a great manner, and this has also made them difficult to compare from before and after cleaning. It was also not taken great enough care to position the samples in the same way as the previous picture. This should however not be a problem as long as the viewer is aware of this. The variations in the angles of the lighting, may influence the way the pictures are compared. However, it is unlikely to affect the overall results, as the results for the colour change are based mainly on the colorimetry results, and not on the visual observations of the samples.

Another way of distinguishing colour change is by calculating  $\Delta E^*$  ( =

 $\sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}$  ) (Witzel, Burnham, & Onley, 1973; Wyszecki & Fielder, 1971). This is a method in which the differences in  $L^*$ ,  $a^*$  and  $b^*$  before and after cleaning, are calculated, to evaluate the actual change in colour by a numerical value. This value indicates the degree of colour change, but it does not as in the previous values indicate which colours have been changed (Konica-Minolta, 2003, p. 15). All the  $\Delta E^*$  results can be viewed in Appendix 4. The numbers given below are based on SCE values.

*Table 4: Values for*  $\Delta E^*$  (*Mokrzycki & Tatol, 2011, p. 15*).

0 – 1	Observer does not notice a difference
1 – 2	Only experienced observer can notice the difference
2 - 3.5	Unexperienced observer notice the difference (Perceivable)
3.5 – 5	Clear difference in colour
5 AND ABOVE	Observer notice two different colours

#### $\Delta E^*$ (CIELAB)

For the samples that were put through the weathering chamber, there were two samples with significant change in the t values. As for the  $\Delta E^*$  values it has a span between 1.51 and 4.24 in the 12 samples. Two of the samples have values between 3.5 and five, which is considered a clear difference in colour.

The samples cleaned with Baolin had two samples that had a significant change in t values. When looking at the  $\Delta E^*$  values they span between 0.89 in sample BA.AS2 to 4.99 in BA.NS3. In  $\Delta E^*$  4.99 is considered a significant colour change. BA.NS1 and BA.AS3 which had a significant change in the t values, had a  $\Delta E^*$  value between 2-3.5 which is a perceivable colour difference.

For the samples cleaned with Fulgentin there were two samples that had a significant colour change in t values. In  $\Delta E^*$  it spans from 0.26 in FU.NS2 to 5.45 in FU.FS2. 5.45 is a number indicating that the sample has gotten a different colour, and this also belong to a sample that had a significant change in the t values. The other sample FU.FS1 that had a significant change in t values as well had a  $\Delta E^*$  value of 2.86, this is considered a perceivable colour difference.

The samples cleaned with Centurio had as much as 8 samples with significant change in the t values. As for the  $\Delta E^*$  values, it has 7 samples with numbers above 5, and numbers above five indicate that the colour has changed to such a degree that it can be considered a different colour. This also includes CE.AS1, which did not have a significant change in the t values. The two other samples CE.NS1 and CE.NS2 have numbers that indicate a perceivable colour change.

After having calculated  $\Delta E^*$  for the samples, this show that there has occurred colour change on the samples that is visible. And there are even eight samples that have had such a big change to their colours that it can be considered a new colour. As this is a more scientific way of calculating the visible colour change this is more reliable than the evaluation of the pictures.

#### 4.2.6 Residues

As discussed above there is a possibility that there might be residues on the cleaned Centurio samples. This might also have affected the results concerning colour measurements. If the cleaning product is lying like a layer on top of the shellac, this can distort the original colours. The cleaning product has its own colour, this might have affected the colour measurements after cleaning. As changes has mostly happened with the Centurio cleaned samples, this show the same trend as with the FTIR results.

# 4.2.7 Have the results been affected by the intrinsic variation of colour of the wood?

When investigating the colour change on the samples cleaned with Centurio, it is important to consider whether these results has been influenced by other factors. One factor that could have influenced the changes is the large colour variation of the wood, which is still visible after the shellac was applied. However, as mentioned in chapter 2, the wood samples were assigned randomly into the different categories, so to avoid biases under this point of view.

In addition, 20 measurements were taken in each step of the study of every sample. This was done to ensure that the results would not be influenced by local variations. These measurements were taken to give the "complete picture" of the colour of each sample. The use of the Grubbs' test excluded outlier values at the 95% confidence, thus supporting the quality of the colorimetric results, meaning the colours that are too light or too dark and thus could shift the whole colour values drastically, have been excluded. Because of this, the chances that the uneven colouring have influenced the colorimetry results, are slim.

### 4.3 Gloss measurements

#### 4.3.1 Summary of gloss measurements

When reviewing the gloss measurements for all of the samples, they show the same inconsistency. The three persons who evaluated the results had much difficulty in evaluating whether there had been a change between the different stages of the samples. There was also a difference in opinion for many of the samples, meaning that no real conclusions could be drawn from these results. The only place where it could be distinguished a change was on the aged samples. Here, it was possible to perceive a difference between the area covered by the metal stand and the aged part of the samples. This difference could be considered as a loss of gloss, as the aged part of the samples have a more matte appearance. No visible change in gloss of the samples after cleaning was perceived. This is important, as all of these products claim to give a glossier aspect to the treated surfaces (A. B. Ernst P; A. B. Ernst P; MAPA).

A highly glossy surface is often something that is desired in a piece of furniture, and alterations in gloss can destroy the desired look that the furniture should have (Allen, 1994, p. 24). When considering shellac, gloss is one of the factors that can be altered due to chemical changes. This makes the evaluation of the gloss important to get the whole picture of the effect these three cleaning products have on the shellac coating. It is therefore unfortunate that there are no significant results to be gained from this method.

#### **4.3.2** Considerations on the method

The method used for evaluating the gloss of the samples is a quite simple one, and something that can be done without a lot of resources. It is based on a direct visual evaluation of the

gloss or by taking pictures of the surface. This method is a very subjective way of evaluating gloss, as no set parameters for the different categories of the gloss are available. This will lead to people perceiving differences in different ways. Following this, it was difficult to decide how to approach the evaluation of the mock-ups used in this thesis. As they are not very glossy, the reflection of the patterns did not show very well on the samples itself or the pictures, resulting in insignificant results. Therefore, it would be ideal to have used another method for evaluating possible changes in gloss of the mock-ups.

A common way of investigating gloss is by using a glossmeter (Gupta, Singh, & Kishan Kumar, 2016, pp. 94-95; Kubick & Giaccai, 2012, pp. 45-50). A glossmeter is an instrument in which it can be decided at which angle the gloss is evaluated, and determines the intensity of the reflection of light (Elcometer, 2018). Such an instrument would have been able to provide in percentage a possible (even not perceivable) loss of gloss of the surface (Gupta et al., 2016).

#### 4.3.3 Not polished samples

As briefly mentioned before, French polishing is a type of finishing that gives furniture a high gloss (St Leger Kelly, 2006, p. 153). In this work, the mock-ups were not polished after the application of shellac. Therefore, their gloss is not as pronounced as it would be desired for shellac furniture. The lack of polishing has certainly affected the gloss level of the samples. Even though the samples did not have high gloss surfaces, it could have been possible to detect changes in the gloss that are there. This would however require a better analysis method than the one used in this work, such as a gloss meter, mentioned above. If such a method was used, the differences would be easier to distinguish. As described in Allen this final polish gives the surface a toughness as well as a high gloss surface. This means that the surface can be more withstanding than an unpolished surface, meaning that it is more resistant to scratches which is one of the factors that can distort gloss. When considering this factor non-polished samples are more likely to lose gloss than a polished sample, because it lacks the hardness of the polished surface. Therefore, the lack of polishing may have influenced the gloss measurements of the samples. However, because of the inconsistency in the results, it was impossible to draw any conclusions from the gloss measurements and it has not been of any consequence to the interpretation of the results.

# 4.4 Conservation aspects

#### 4.4.1 Representativity of the samples

This study has been performed to investigate different cleaning products that could possibly be used for cleaning the furniture at the royal collections of Norway, with a goal to find a safe method for cleaning this furniture. As this work has used mock-ups rather than the actual furniture in question, it is important to consider whether these mock-ups are representative of the actual historical furniture.

#### Wood

The wood used for the mock-ups, mahogany, was chosen based on conversations with the conservator and carpenter at the royal collections, as parts of their furniture collection is made from mahogany wood (Schiander & Skipperud, 2017). The mahogany used for this research was purchased in 2017. In later years, the trade and production of exotic woods such as mahogany has been restricted, because of the preservation of the rainforests. Because of these restrictions of availability, investigations into what kind of mahogany was used for the original furniture were not carried out. But even if the wood is of different origin, the different mahogany woods are quite similar in texture and it is doubtful that these differences would have influenced the results (Edlin, 1977).

Another problem relating to the use of this wood was that it was a relatively fresh one, directly acquired from a carpenter. This meant that the samples have not dried completely, as the wood used for furniture often had been. When the samples were weighed after the application of shellac, they had lost weight rather than gaining it, this can be an indication that the samples were not dry before the coating of shellac (Appendix 1). Even though the samples were not completely dry, the chances for this to influence the representativity of the samples are minimal.

#### Polishing

According to Allen, the method for French polishing of furniture is; many layers of shellac bonded together followed by a polishing of the surface (1994, pp. 34-46). This final polish gives a high gloss and a toughness to the surface. French polishing would have been the method for how the shellac furniture at the royal collections were made. In spite of this, the mock-ups created for this thesis were not polished after the application of the shellac, and the number of layers of shellac on the samples is probably too few compared to the historical varnishes. The decision not to polish the samples after application was taken on the basis of practical feasibility of this thesis work. First of all, polishing a surface requires a bigger area than the one of the mock-ups (Hagen, 2017). Secondly, polishing each single sample would have required a lot of time, and the result would however not have been optimal compared to the furniture. The difference in the non-polished samples compared to the polished samples is - as outlined above - in the highly glossy surface and its toughness. Therefore, it is likely that this aspect affects the representativity of the samples. In regard to the analysis performed to investigate the changes in chemical composition, this is not likely to have affected the results. The chemistry of shellac is the same whether the surface that has been varnished with it has been polished or not. Therefore, when testing changes in the chemistry, it was not imperative to polish the samples. It is likewise improbable that the colour of the samples would have been substantially different from the one of polished surfaces.

#### Ageing

Considering ageing for the representativity of the samples with respect to the actual furniture is challenging. Some of the furniture in question has been acquired at different times and has been subjected to different deteriorating factors, affecting each piece of furniture differently. Here, it is also important to mention that the furniture in question might have been restored during the years, leading to a different pattern in the ageing of the object. Considering this, the ageing of the furniture can vary within the different pieces of furniture, it would be difficult to set a certain deterioration for all the samples based on the furniture itself (Caple, 2000, pp. 70-78). To assess the level of weathering to the real furniture would have also taken a lot of time for testing, which was deemed unnecessary for the tests performed in this study.

#### 4.4.2 Application of cleaning products

When evaluating cleaning products, one of the key elements that need to be recognized is the way the cleaning products have been applied. Different application methods can result in different cleaning results. Thus, it is necessary to look into how the application of the cleaning products could have altered the results obtained in this thesis.

#### **Application time**

As described in chapter 2, the method for how long to apply the cleaning products was derived from the instructions of each of the cleaning products. These gave approximately the same directions about application time. The instructions for Baolin, however, also stated that, if the dirt on the treated surface had been quite resistant, it could be necessary to leave the cleaning product on the surface for multiple hours. This was only mentioned as a possibility for Baolin, and not for the others two products. Considering that these cleaning products all contain different chemicals, the exposure time with shellac could make a difference in the effect they had on the resin. Even though the instructions mention only short exposure times for two of the cleaning products, there is still the possibility that they also might be used for longer periods of time. Taking into account that the exposure time might be prolonged, by for instance an hour for all the cleaning products, the alterations in the physico-chemical properties of shellac can be more pronounced than the effects observed in this thesis, or different altogether. Another issue arising when having a prolonged exposure time can be that the cleaning product can be harder to remove from the surface, since the cleaning product might somehow dry on the surface. It could then be necessary to use a moistened cloth to get all the cleaning product off the surface. As shellac and water do not react well, the need to use water to remove the cleaning products would work against the cleaning products intentions

#### **Application method**

Another important aspect is the application method. How the product is applied can affect at least the surface of the samples. Different application techniques, such as circular movements or straight movements, can, for instance, affect the gloss by micro-scratching the surface. When it comes to these cleaning products, no specific application methods are provided other than the application with a cloth, and its subsequent removal from the surface. This gives an uncertain factor, as the movement in which to apply the product is open to the choice of the

conservator. As this thesis aimed to have as little variables as possible, the same application method was employed for all cleaning procedures: to apply the product in a straight manner, following the fibres of the wood. Making such a decision was necessary to exclude other methods, but it is also not given that this is the optimal way of applying the products. Therefore, different methods of application should be tested in the future. Another factor (yet very difficult to keep into account) related to the application method is the pressure used when applying the cleaning products. All the cleaning products probably have some form of abrasive in them. Therefore, the more pressure is applied, the more the surface varnished with shellac could be affected. The type of cloth can also be a variable when cleaning with these products. As the cloth can have varying degrees of softness and fibres in them, it is possible that different kinds of cloth can cause different damage, such as abrasion on the surface. Therefore, it is important to consider carefully what type of cloth is used during the cleaning.

#### 4.4.3 Non-invasive methods

Scientific analysis has always been playing a great role in conservation practice, and the methods have changed with the professional trends. These have been greatly affected by the ethical codes that have been developed since the 1960s with the Murray Pease report and the Venice charter (Sease, 1998, pp. 98-100). Following this first establishment of ethical codes, many others followed. As these codes of ethics have gradually been established, the terms "minimal intervention" and "non-invasive techniques" have been implemented in conservation practice.

*Minimal intervention* gained momentum in the 1980s, and has thereafter been recognised as a key point within conservation (Caple, 2000, pp. 64-65). Minimal intervention is the concept of altering or adding as little as possible to the object, after having considered it carefully before the action.

*Non-invasive techniques* are analytical methods that do not remove anything from the object. And since the concept of minimal intervention is aiming at altering the object as little as possible, non-invasive techniques are in line with this.

These terms are now some of the most important to consider before doing material analysis on objects, and it is always an aim to use all possible non-invasive techniques before going to the step of removing samples from historical objects.

In this work, only non-invasive methods have been employed to obtain the results with the exception of the varnish thickness measurements. These methods were in line with the ethical guidelines in regards to minimal intervention. All the measurements were focused on investigating the surface of the samples, hence not requiring samples to be removed from any of the samples. None of the employed methods has done any damage to the measured areas of the samples in this process.

An alternative to the methods used for this work would be invasive or micro-destructive methods. These methods require sampling that are destroyed during the analytical process. Such methods could provide a more detailed account of the chemistry of the coating and give a clearer idea of what the effects of the cleaning products are. FTIR is considered one of the best ways of investigating polymers (Bradley, 2018). In this thesis, however, the most recent development of this technique (handheld FTIR) was used. Another way of investigating the varnish could be a laboratory FTIR (Stuart, 2007, pp. 110-111). This method does require a sample of the varnish layers, however, it is a more established method and can provide better measurements on the samples.

If it was desirable to investigate the substance and effects more closely, different chromatographic methods could be employed. These are methods used to separate mixtures and they can detect small amounts of component (Stuart, 2007, pp. 296-297). Therefore, they can be useful in analysing samples of historic objects. There are different methods for using chromatography such as gas chromatography or high performance liquid chromatography, these are often coupled with a mass spectrometer which can identify the substances.

Considering that the test objects used in this thesis are mock-ups and not historical objects, it would be possible to use invasive methods for analysis, since they are not covered by the ethical guidelines in the same way as historical objects are. Therefore, this would be a good opportunity to employ micro-destructive techniques that generally lead to a high amount of information. Creating mock-ups, such as in this work, is a common way of investigating different conservation treatments for cultural heritage. It was however decided to use only non-invasive methods for this work. This was also based on the wish to create a way of investigating the coating on historical furniture. This method would have to be in line with the ethical guidelines for conservators, and such be based on minimal intervention with the

original materials. Considering this and the fact that the results have been of high quality, this method of analysing coatings can be considered a success. This method can be considered as a starting point for creating a protocol for non-destructive investigation of cleaning products.

# **5** Conclusion

This chapter will gather the most important findings of this thesis and focus on some of the further research that should be done on this topic.

# 5.1 Summary

To conclude, this thesis has been able to provide light on some of the effects certain cleaning products has on shellac. It has also been possible to do this study based on a non-invasive approach.

#### 5.1.1 Summary of cleaning tests

The FTIR results of the cleaning test with Baolin and Fulgentin, show that there are no changes in the ER FTIR spectra, after the cleaning process. This is in line with the colorimetry results, where the shellac surface of these samples did not have a significant colour change. There were some samples that had significant colour change but these were not, of such a number that they can indicate a change based on the cleaning.

On the other hand, the samples cleaned with Centurio showed a change in the ER FTIR spectra, after the cleaning process. This difference occurred mainly in the fingerprint region. As this change occurred only on the samples cleaned with Centurio, this indicate that it is the cleaning product that causes these changes. The changes in the spectra can be either an alteration in the chemical composition of shellac, or it can be an indication that there are residues of Centurio on the surface of the cleaned samples. Following the FTIR results, were the colorimetry results which supports the findings in the FTIR results. These results show that as much as eight of nine samples cleaned with Centurio, had a significant colour change after the cleaning process.

One of the aims for the thesis was to investigate whether there were any differences in the effect of the cleaning products, between freshly applied shellac and artificially aged shellac. When looking at the samples cleaned with Centurio, the different categories react in the same way, the same goes for the samples cleaned with Baolin. The only note that can be made on

this point is that in the colorimetri data for Fulgentin, only FS samples changed after the cleaning. Otherwise there does not seem to be any difference between the aged shellac or the fresh shellac in the way it reacts to the cleaning products.

#### 5.1.2 Non-invasive approach

During the work on this thesis a non-invasive approach for investigating coatings has been developed. This method could without sampling detect alterations in the physico-chemical properties of the coating. Therefore, this approach can be employed to investigate coatings on furniture without interfering with the original materials.

### 5.2 Further outlook

There is at this stage of the research, no grounds for considering the cleaning products suitable for cleaning shellac varnish. Therefore, there are many ways of taking this research forward, some of which are quite important to understand fully the results obtained in this thesis.

As described earlier, this study was done over a very short timeline. Even though the testing period was limited, some of the results indicate that there was enough time for the cleaning products to react, as Centurio did with the shellac coating. There is however a possibility that with a long term study, there can be gained a greater knowledge of the reactions with the different cleaning products. This would give a better insight into the long term effects of all the cleaning products on a shellac coating. It is also important to be able to rule out any possible long term effects Baolin and Fulgentin might have on the shellac coating, that was not discovered in this study. Also, for Centurio a long term study would be beneficial as it should be investigated whether the negative effects it had on the shellac, would be greater over time.

For a better understanding on why Centurio has such an effect on the shellac coating compared to the other cleaning products, it would be important to investigate the cause. As the material knowledge of each of the cleaning products is only based on the commercial information found in their datasheet, the cause of these physico-chemical changes is unknown. To fully understand these changes, a full chemical analysis of each cleaning product should be conducted, to provide a more detailed account of the constituents of them.

Considering that this thesis only has focused on the physico-chemical effect these cleaning products have on the shellac coating, it is important to point out that there are many other considerations that need to be met for a cleaning product to be suitable for historical objects. One of these factors is the cleaning effect these products have on the shellac. If a cleaning product causes no physico-chemical changes on the shellac, but at the same time it does not provide a suitable cleaning effect, this could not be considered a good cleaning product, as it cannot fulfil the designated task set for the product. It is therefore important to broaden the spectre of criteria in which the cleaning products are tested in further studies. Only considering one of the factors, as has been done in this thesis, is just a starting phase of the survey of these products.

As described above this thesis has been narrowed into a small portion of the greater picture and there are ways of broadening the research. Not only the criteria for testing but also the materials for testing need to be altered, such as the type of wood as well as the type of shellac tested. Altering the materials that are tested should be a good indicator for whether the cleaning products react differently to different types of shellac or wood.

When some of the ideas for further research, as mentioned above have been done, the next step should be to test the cleaning products on real historical shellac varnish. As this varnish will have deteriorated in a more unpredictable way than the aged mock-up samples used in this thesis it could give a different result.

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# 6 Appendix

# 6.1 Appendix 1:

Sample	Length in cm	Width in cm	Thickness in cm	Weight before shellac	Weight after shellac
CO. FS 1 Average	15.30 cm	7.12 cm	1.52 cm	94.26 g 94.26 g <u>94.26 g</u> <b>94.26 g</b>	93.30 g 93.29 g <u>93.28 g</u> <b>93.29 g</b>
CO. FS 2 Average	15.42 cm	7.12 cm	1.52 cm	93.49 g 93.49 g <u>93.50 g</u> <b>93.49 g</b>	92.59 g 92.58 g <u>92.59 g</u> <b>92.59 g</b>
CO. FS 3 Average	15.44 cm	7.12 cm	1.54 cm	94.09 g 94.08 g <u>94.08 g</u> <b>94.08 g</b>	93.08 g 93.09 g <u>93.08 g</u> <b>93.08 g</b>
CE. FS 1 Average	15.27 cm	7.14 cm	1.52 cm	97.18 g 97.18 g <u>97.18 g</u> <b>97.18 g</b>	96.18 g 96.18 g <u>96.18 g</u> <b>96.18 g</b>
CE. FS 2 Average	15.26 cm	7.14 cm	1.52 cm	87.45 g 87.45 g <u>87.44 g</u> <b>87.44 g</b>	86.69 g 86.69 g <u>86.68 g</u> <b>86.69 g</b>
CE. FS 3 Average	15.41 cm	7.14 cm	1.52 cm	92.90 g 92.90 g <u>92.90 g</u> <b>92.90 g</b>	91.91 g 91.91 g <u>91.91 g</u> <b>91.91 g</b>
BA. FS 1 Average	15.08 cm	7.13 cm	1.52 cm	84.23 g 84.23 g <u>84.24 g</u> <b>84.24 g</b>	83.48 g 83.47 g <u>83.47 g</u> 83.47 g
BA. FS 2 Average	15.30 cm	7.14 cm	1.54 cm	97.00 g 97.00 g <u>97.00 g</u> <b>97.00 g</b>	95.80 g 95.79 g <u>95.79 g</u> <b>95.79 g</b>
BA. FS 3 Average	15.10 cm	7.10 cm	1.54 cm	86. 05 g 86.06 g 86.06 g 86.06 g	85.22 g 85.22 g 85.21 g 85.22 g
FU. FS 1	15.50 cm	7.15 cm	1.54 cm	92.46 g 92.46 g 92.47 g	91.61 g 91.62 g 91.61 g

Average				92.46 g	91.61 g
FU. FS 2	15.30 cm	7.15 cm	1.52 cm	95.15 g	94.21 g
101182	10.000	,		95.16 g	94.21 g
				95.16 g	94.22 g
Average				95.16 g	94.21 g
FU. FS 3	15.61 cm	7.14 cm	1.54 cm	91.41 g	90.48 g
10.155	15.01 011	7.1 <b>4 C</b> III	1.54 611	91.42 g	90.48 g
				91.42 g	<u>90.48 g</u>
Avorago					
Average CO. AS 1	15.31 cm	7.14 cm	1.54 cm	91.42 g	90.48 g
CO. AS I	15.51 cm	/.14 cm	1.54 cm	90.28 g	89.53 g
				90.29 g	89.53 g
				<u>90.28 g</u>	<u>89.54 g</u>
Average	1.5.01	- 12	1.50	90.28 g	89.53 g
CO. AS 2	15.31 cm	7.13 cm	1.53 cm	91.33 g	90.58 g
				91.32 g	90.58 g
				<u>91.33 g</u>	<u>90.58 g</u>
Average				91.33 g	90.58 g
<b>CO. AS 3</b>	15.39 cm	7.09 cm	1.54 cm	86.00 g	85.10 g
				85.99 g	85.11 g
				<u>85.99 g</u>	<u>85.10 g</u>
Average				85.99 g	85.10 g
CE. AS 1	15.27 cm	7.13 cm	1.52 cm	89.63 g	88.71 g
				89.63 g	88.71 g
				89.63 g	88.71 g
Average				89.63 g	88.71 g
CE.AS 2	15.34 cm	7.14 cm	1.55 cm	93.83 g	92.72 g
				93.83 g	92.72 g
				<u>93.83 g</u>	<u>92.72 g</u>
Average				93.83 g	92.72 g
CE.AS 3	15.34 cm	7.14 cm	1.54 cm	90.12 g	89.36 g
				90.11 g	89.36 g
				<u>90.10 g</u>	<u>89.36 g</u>
Average				90.11 g	89.36 g
BA. AS 1	15.15 cm	7.14 cm	1.54 cm	92.67 g	91.79 g
	10.10 0111	,	1.0 1 0111	92.68 g	91.79 g
				<u>92.67 g</u>	91.79 g
Average				92.67 g	<u>91.79 g</u>
BA. AS 2	15.21 cm	7.15 cm	1.55 cm	88.32 g	87.32 g
	13.21 0111	/.15 CIII	1.55 011	88.31 g	87.31 g
				<u>88.32 g</u>	<u>87.32 g</u>
Average				88.32 g	87.32 g
BA. AS 3	15.41 cm	7.14 cm	1.54 cm	90.91 g	
DA. AS J	13.41 011	/.14 CIII	1.34 CIII	•	90.24 g
				90.92 g	90.24 g
A				<u>90.92 g</u>	<u>90.23 g</u>
Average	16.61	7.16	1.52	90.92 g	90.24 g
FU. AS 1	15.51 cm	7.16 cm	1.53 cm	94.36 g	93.40 g
				94.36 g	93.41 g
				<u>94.35 g</u>	<u>93.40 g</u>
Average				94.36 g	93.40 g

FU. AS 2	15.40 cm	7.15 cm	1.53 cm	93.27 g	92.28 g
				93.28 g	92.29 g
				<u>93.27 g</u>	<u>92.29 g</u>
Average				93.27 g	92.29 g
FU. AS 3	15.39 cm	7.14 cm	1.53 cm	87.03 g	86.21 g
				87.02 g	86.22 g
				<u>87.02 g</u>	<u>86.22 g</u>
Average				87.02 g	86.22 g
CE. NS 1	14.88 cm	7.11 cm	1.53 cm	88.68 g	85.89 g
				88.68 g	85.88 g
				<u>88.68 g</u>	<u>85.91 g</u>
Average				88.68 g	85.89 g
CE. NS 2	14.86 cm	7.10 cm	1.54 cm	85.07 g	82.44 g
				85.06 g	82.43 g
				<u>85.06 g</u>	<u>82.41 g</u>
Average				85.06 g	82.43 g
CE. NS 3	14.63 cm	7.10 cm	1.53 cm	85.66 g	83.09 g
				85.63 g	83.08 g
				<u>85.62 g</u>	<u>83.09 g</u>
Average				85.64 g	83.09 g
BA. NS 1	14.77 cm	7.11 cm	1.55 cm	85.58 g	82.86 g
				85.59 g	82.87 g
				<u>85.59 g</u>	<u>82.86 g</u>
Average				85.59 g	82.86 g
BA. NS 2	14.85 cm	7.11 cm	1.55 cm	85.52 g	82.90 g
				85.52 g	82.90 g
				<u>85.52 g</u>	<u>82.88 g</u>
Average				85.52 g	82.90 g
BA. NS 3	15.03 cm	7.11 cm	1.54 cm	88.39 g	85.96 g
				88.40 g	85.96 g
				<u>88.39 g</u>	<u>85.96 g</u>
Average				88.39 g	85.96 g
<b>FU. NS 1</b>	14.72 cm	7.11 cm	1.51 cm	83.35 g	80.85 g
				83.34 g	80.85 g
				<u>83.35 g</u>	<u>80.86 g</u>
Average				83.35 g	80.85 g
<b>FU. NS 2</b>	14.95 cm	7.13 cm	1.57 cm	86.58 g	83.90 g
				86.58 g	83.87 g
				<u>86.57 g</u>	<u>83.90 g</u>
Average				86.58 g	83.89 g
FU. NS 3	14.74 cm	7.10 cm	1.54 cm	87.40 g	84.76 g
				87.40 g	84.77 g
				<u>87.39 g</u>	<u>84.76 g</u>
Average				87.40 g	84.76 g
CO. wood 1		7.16 cm	1.56 cm	106.88 g	
				106.86 g	
				<u>106.87 g</u>	
Average				106.87 g	

# 6.2 Appendix 2: Film thickness measurements

Measurements of film thickness with average and standard deviation.

CO.FS1				
Average (7 measurements)	8.85 μm			
Standard deviation	4.07 μm			
Max	14.43 μm			
Min	4.23 μm			
CO.FS2				
Average (6 measurements)	10.02 μm			
Standard deviation	3.95 µm			
Max	16.27 μm			
Min	4.2 μm			
CO.FS3				
Average (7 measurements)	10.42 μm			
Standard deviation	5.61 μm			
Max	22.03 μm			
Min	5.77 μm			
CO.AS1				
Average (8 measurements)	15.09 μm			
Standard deviation	5.23 μm			
Max	21.25 μm			
Min	8.39 μm			
CO.AS2				
Average (6 measurements)	9.88 μm			
Standard deviation	2.96 μm			
Max	13.11 μm			
Min	6.03 μm			
CO.AS3				
Average (6 measurements)	11.16 µm			
Standard deviation	4.77 μm			
Max	21.51 μm			
Min	7.61 μm			

### 6.3 Appendix 3: FTIR spectra

#### **Baolin samples**

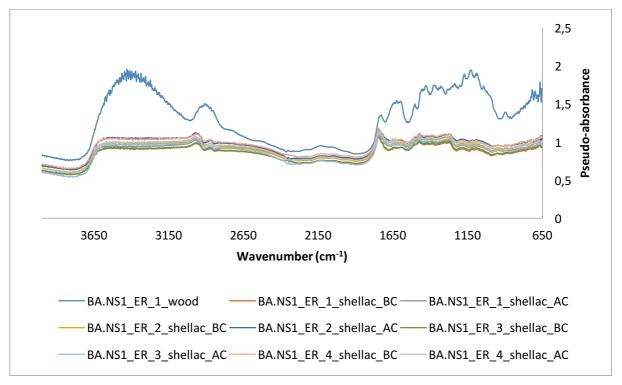


Figure 22: ER FTIR spectra for BA.NS1.

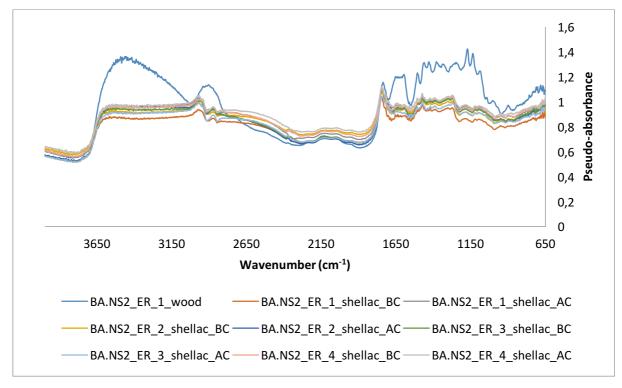


Figure 23: ER FTIR spectra for BA.NS2.

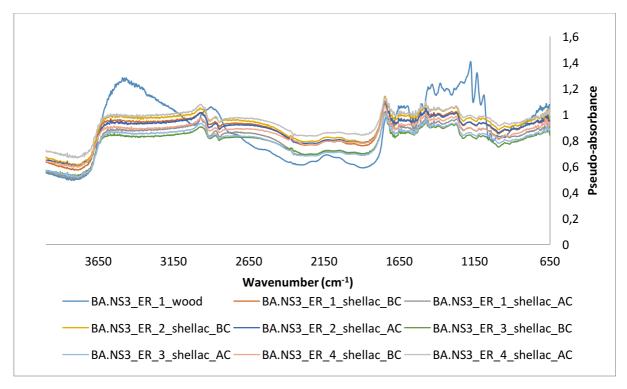


Figure 24: ER FTIR spectra for BA.NS3.

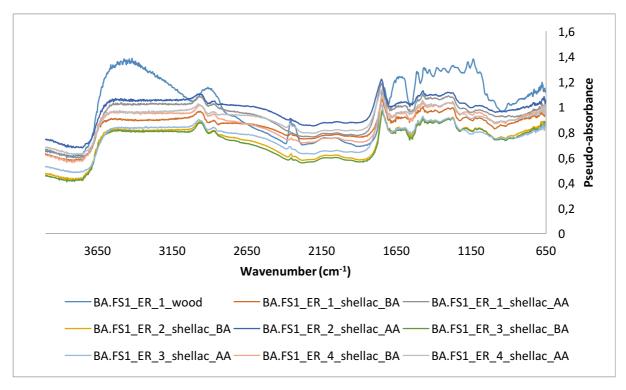


Figure 25: ER FTIR spectra for BA.FS1.

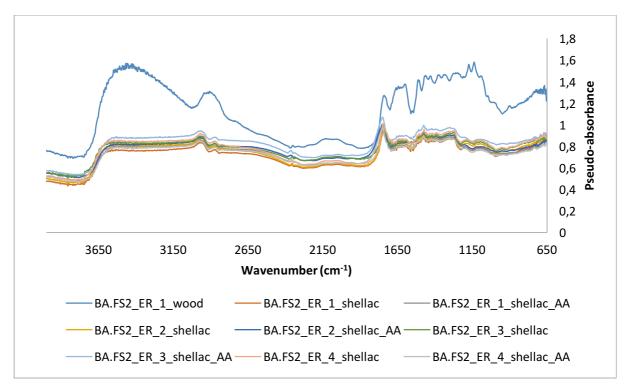


Figure 26: ER FTIR spectra for BA.FS2.

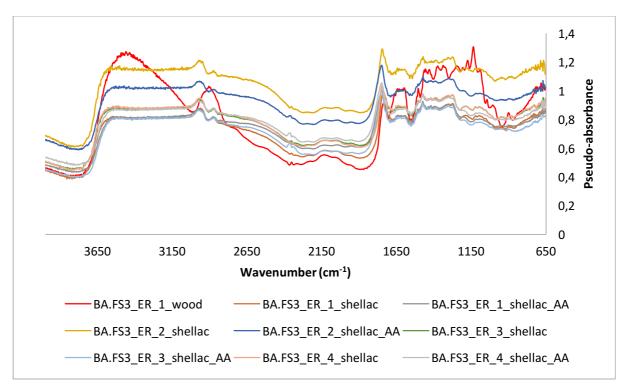


Figure 27:ER FTIR spectra for BA.FS3.

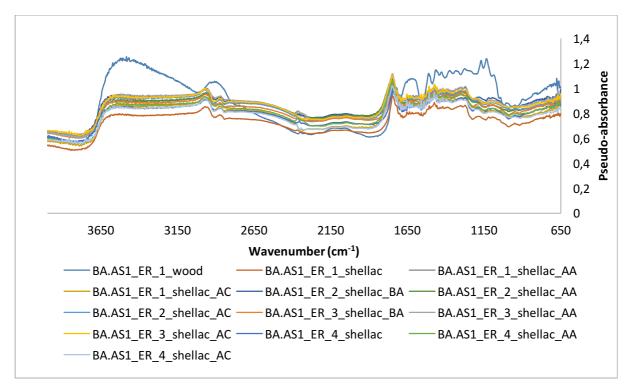


Figure 28: ER FTIR spectra for BA.AS1.

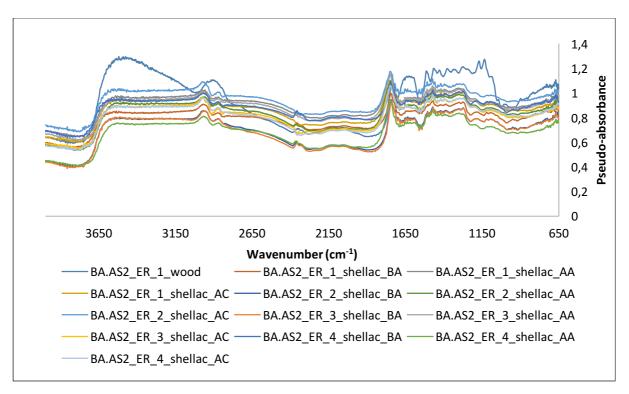


Figure 29: ER FTIR spectra for BA.AS2.

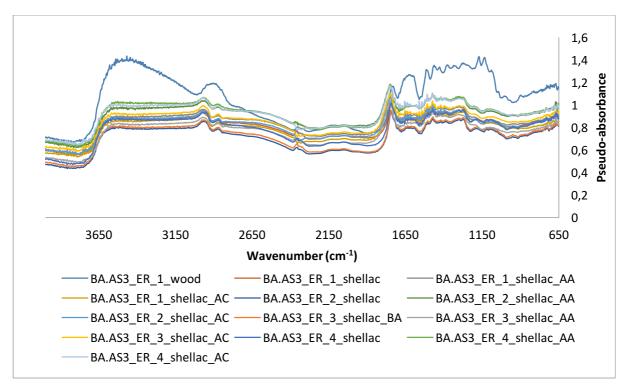


Figure 30: ER FTIR spectra for BA.AS3.

### **Centurio samples**

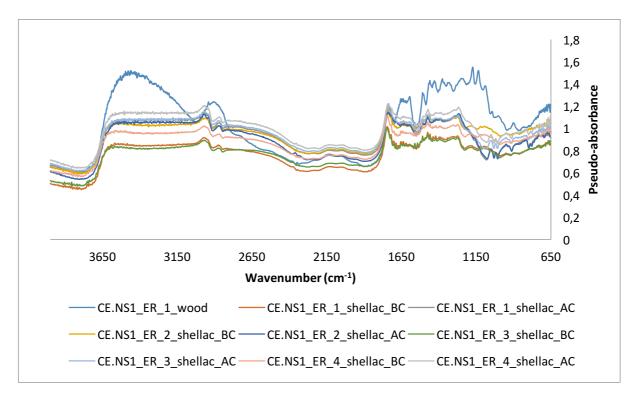


Figure 31: ER FTIR spectra for CE.NS1.

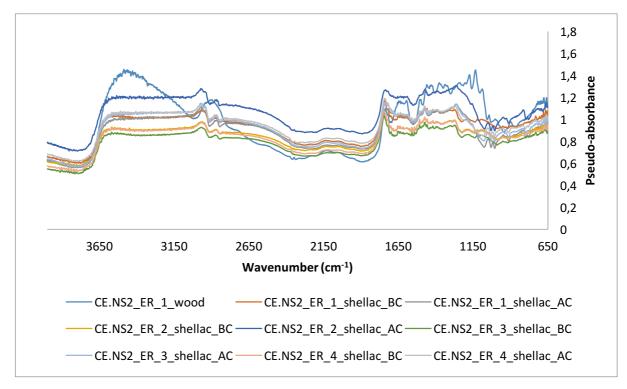


Figure 32: ER FTIR spectra for CE.NS2.

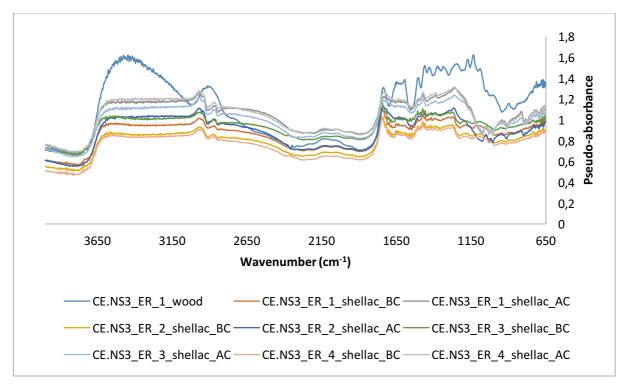


Figure 33: ER FTIR spectra for CE.NS3.

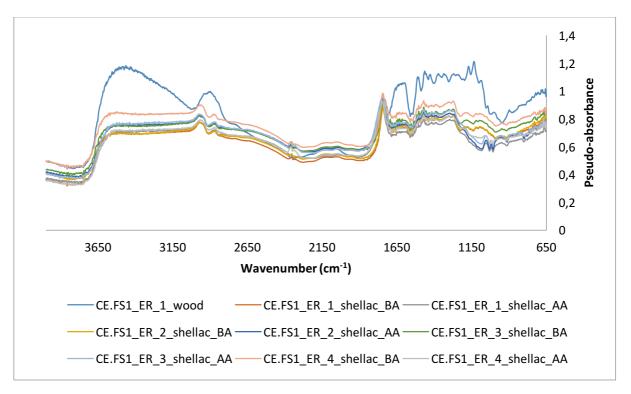


Figure 34: ER FTIR spectra for CE.FS1.

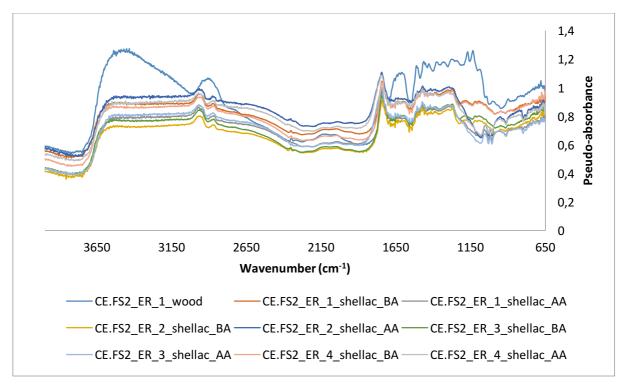


Figure 35: ER FTIR spectra for CE.FS2.

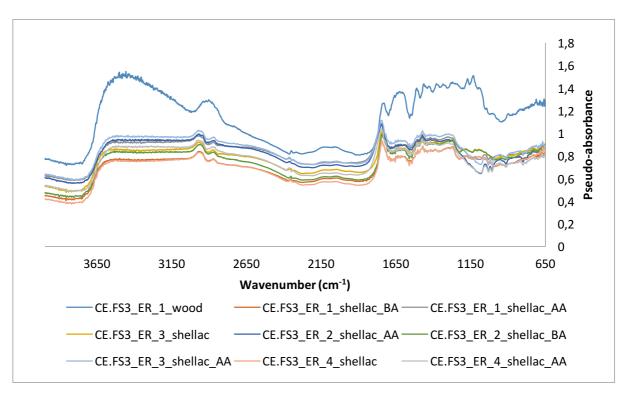


Figure 36: ER FTIR spectra for CE.FS3.

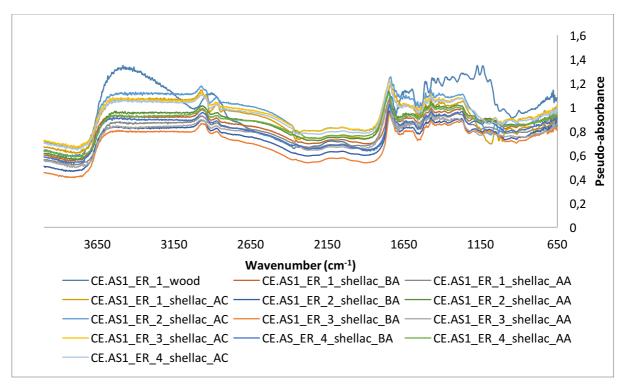


Figure 37: ER FTIR spectra for CE.AS1.

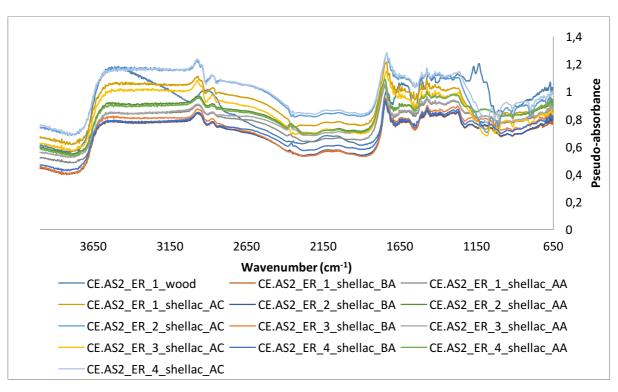


Figure 38: ER FTIR spectra for CE.AS2

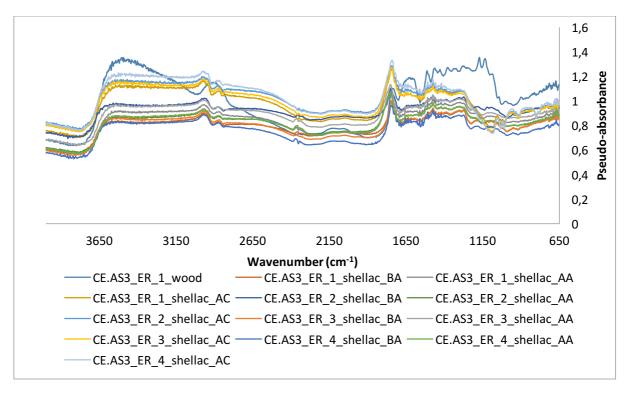


Figure 39: ER FTIR spectra for CE.AS3.

### **Fulgentin samples**

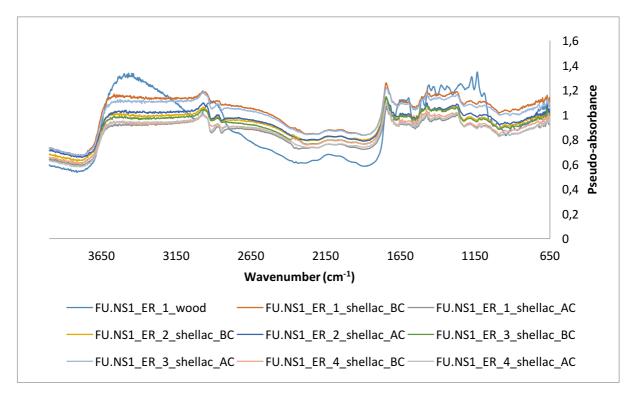


Figure 40: ER FTIR spectra for FU.NS1.

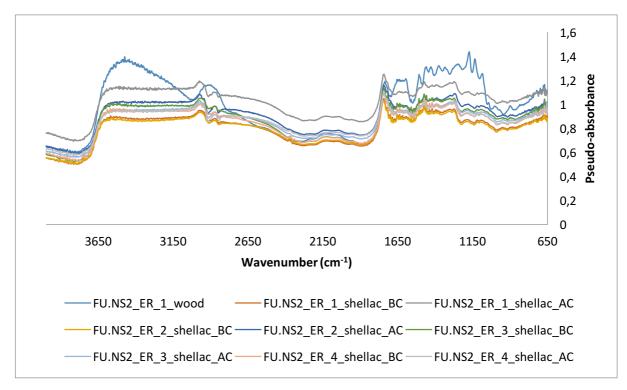


Figure 41: ER FTIR spectra for FU.NS2.

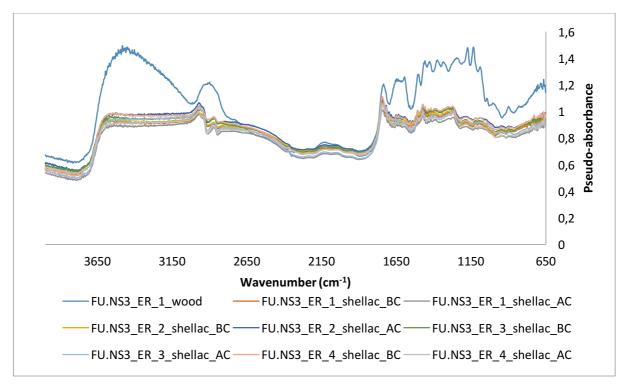


Figure 42: ER FTIR spectra for FU.NS3

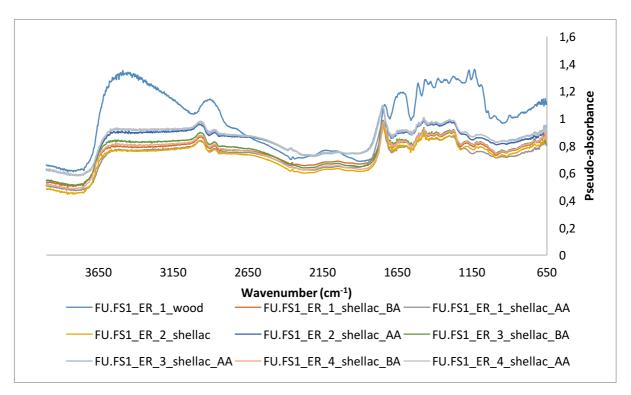


Figure 43: ER FTIR spectra for FU.FS1.

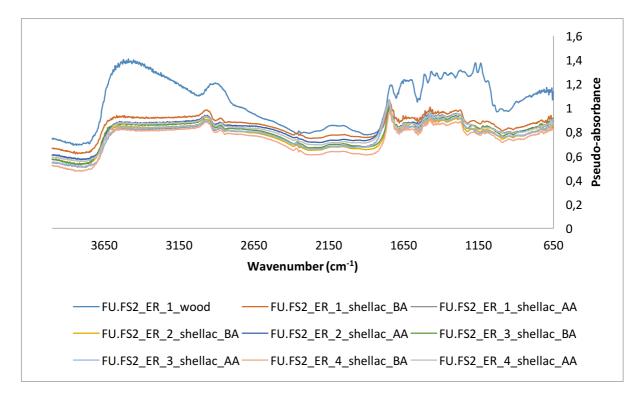


Figure 44: ER FTIR spectra for FU.FS2.

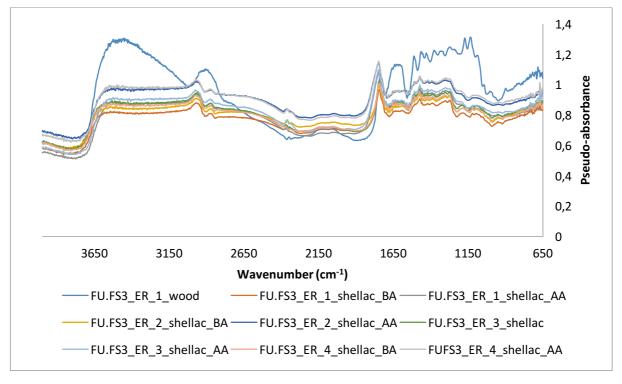


Figure 45: ER FTIR spectra for FU.FS3.

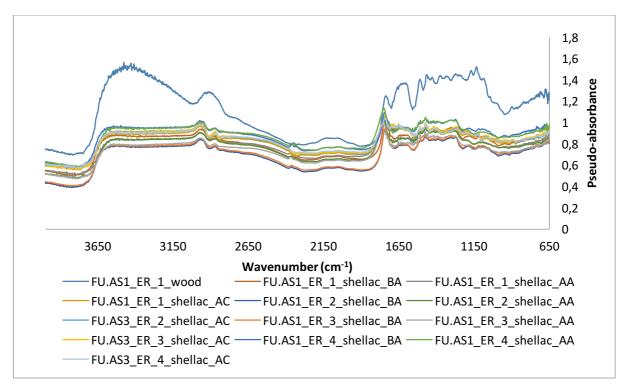


Figure 46: ER FTIR spectra for FU.AS1.

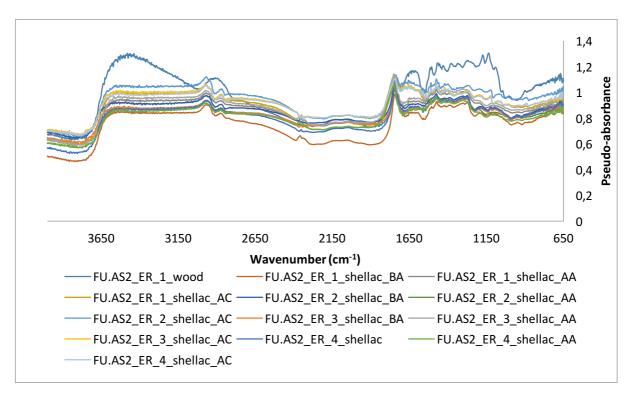


Figure 47: ER FTIR spectra for FU.AS2.

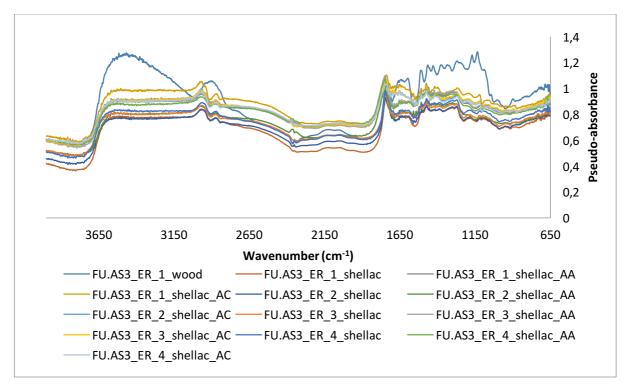
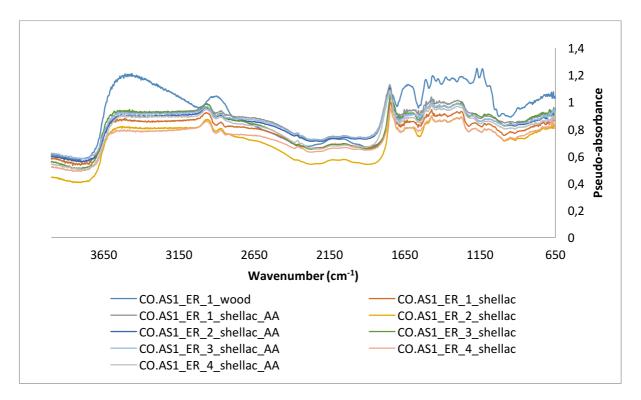
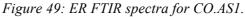


Figure 48: ER FTIR spectra for FU.AS3.

### **Controll samples**





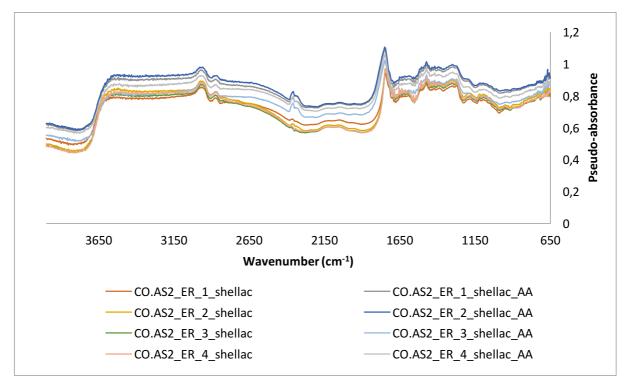


Figure 50: ER FTIR spectra for CO.AS2.

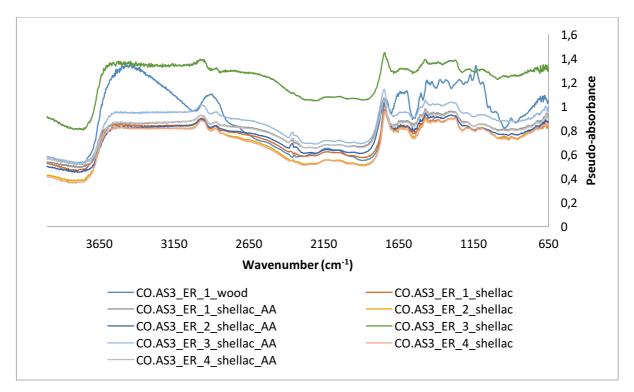


Figure 51: ER FTIR spectra for CO.AS3.

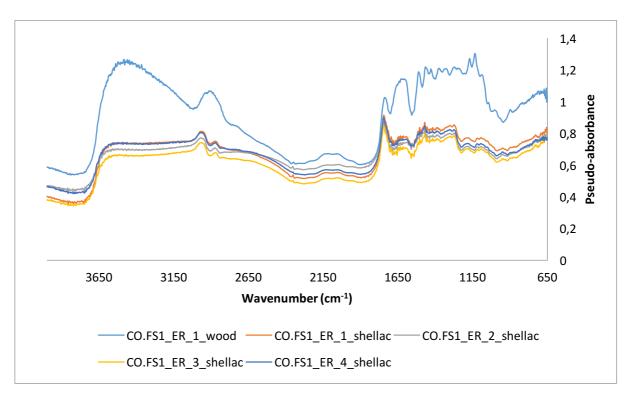


Figure 52: ER FTIR spectra for CO.FS1.

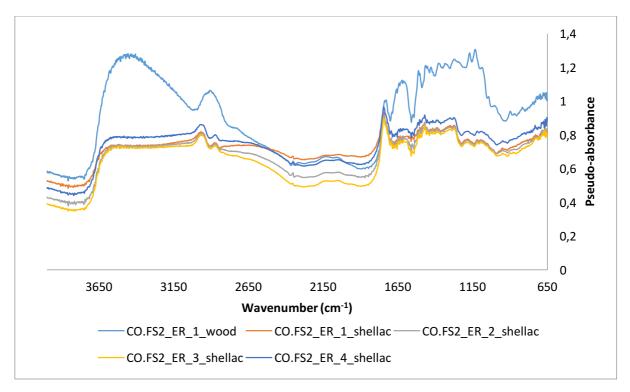


Figure 53: ER FTIR spectra for CO.FS2.

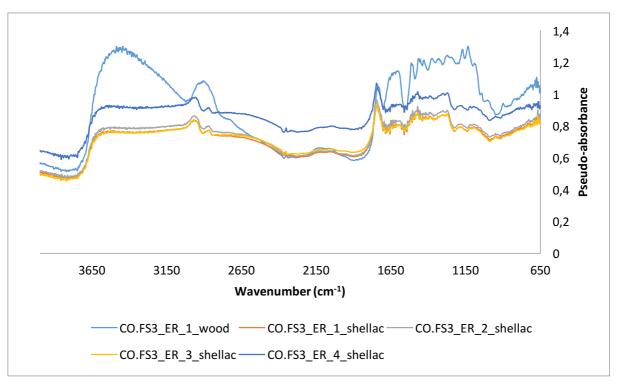
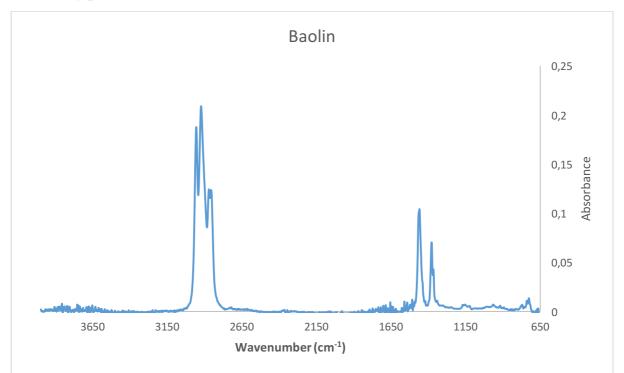


Figure 54: ER FTIR spectra for CO.FS3.

## **Cleaning products**





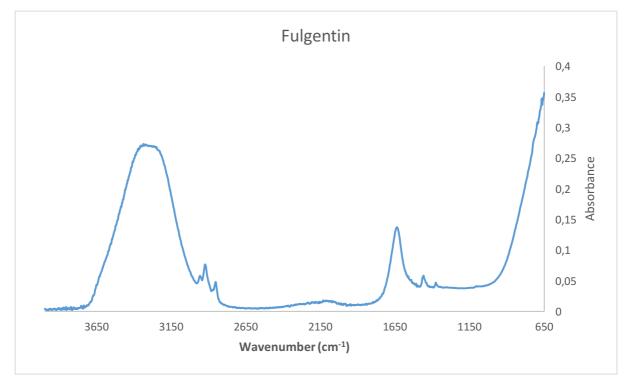
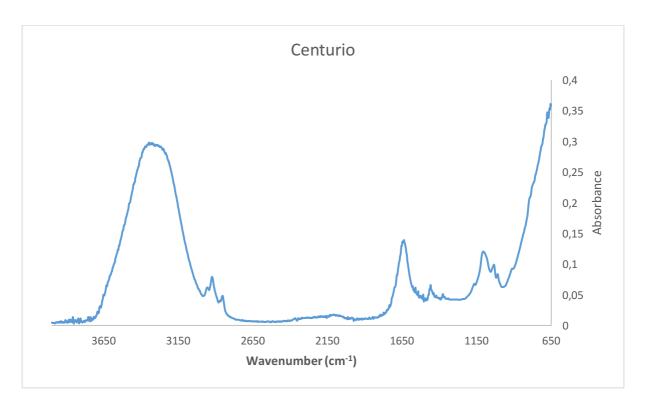


Figure 56: ATR FTIR spectrum of Fulgentin.





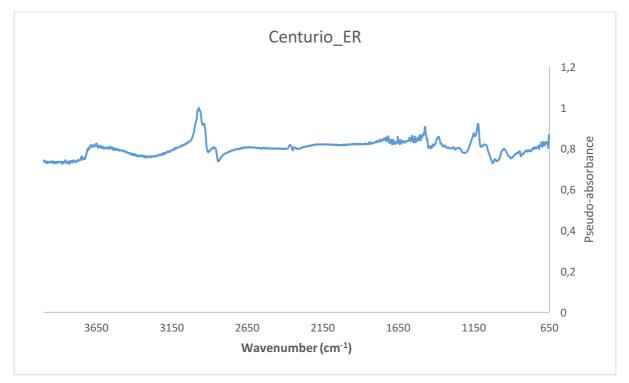
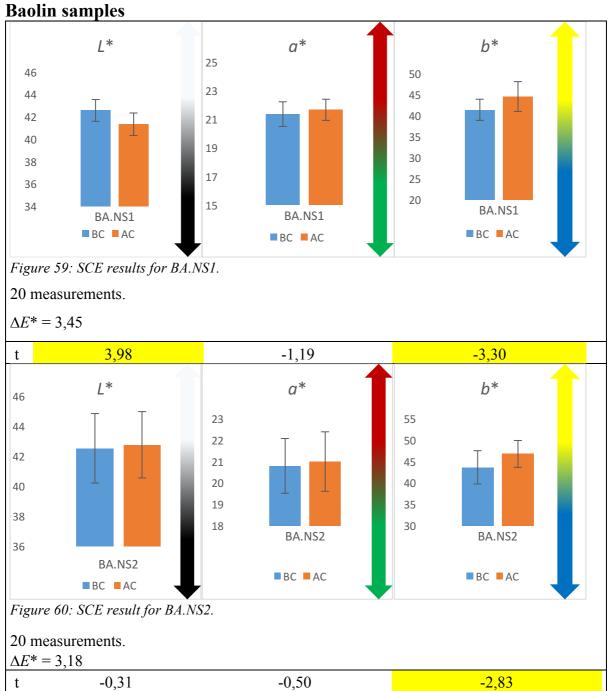


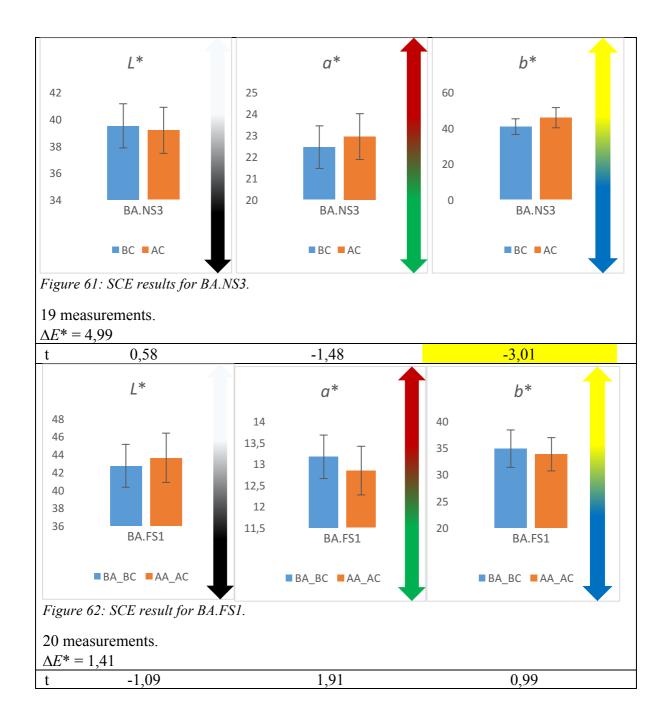
Figure 58: ER FTIR spectrum of Centurio.

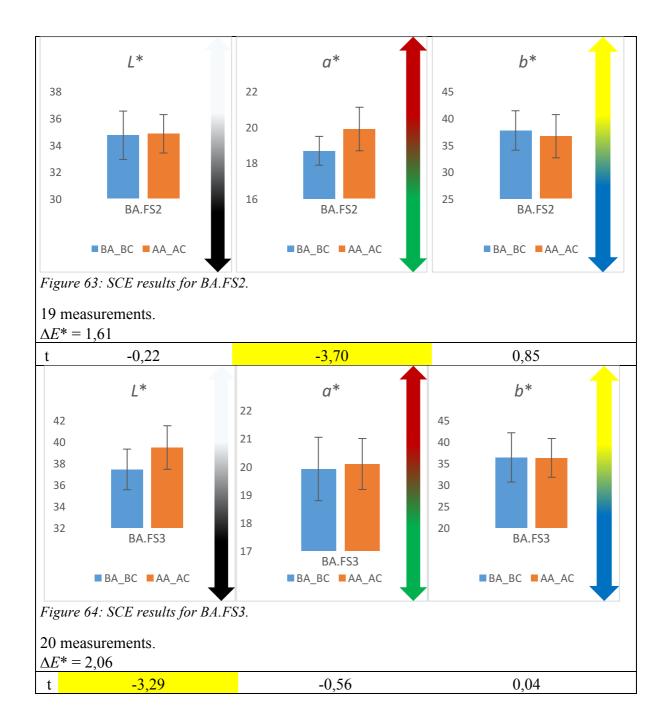
# 6.4 Appendix 4: Colorimetry results

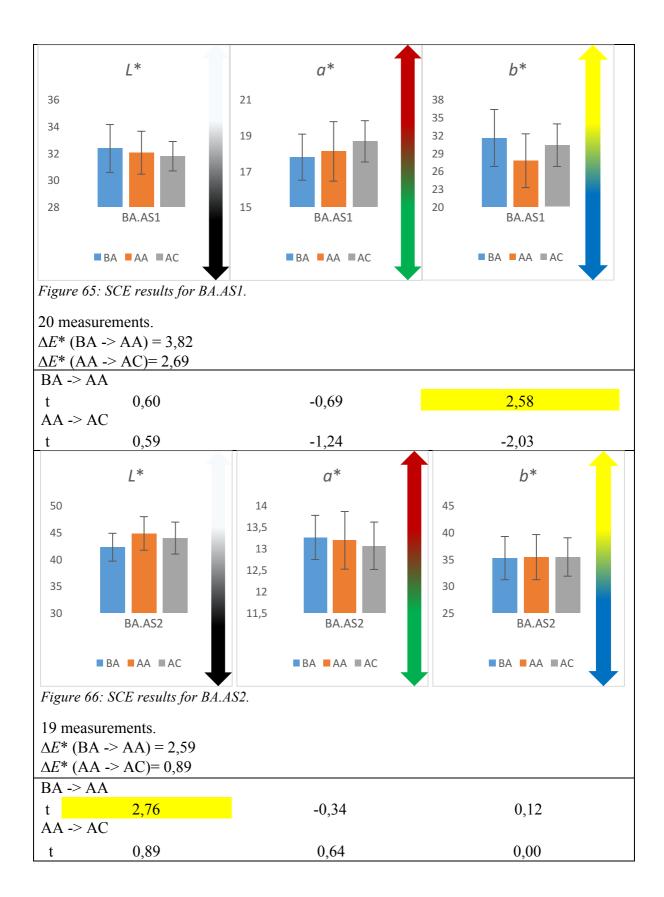
The yellow markings represent a value that is above t critical value, and therefore considered a significant change in colour.

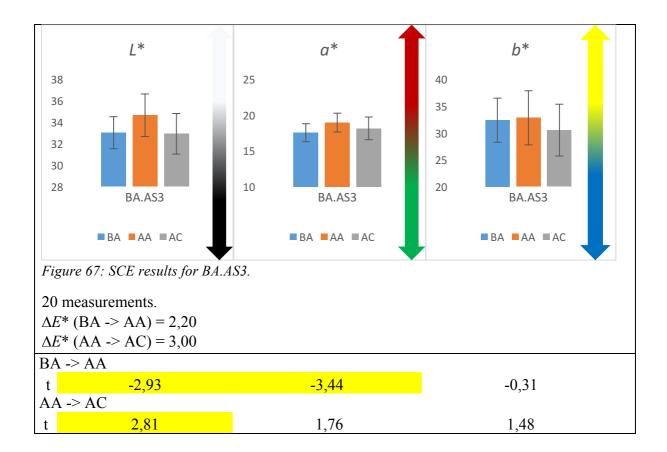


#### SCE results Baolin sample

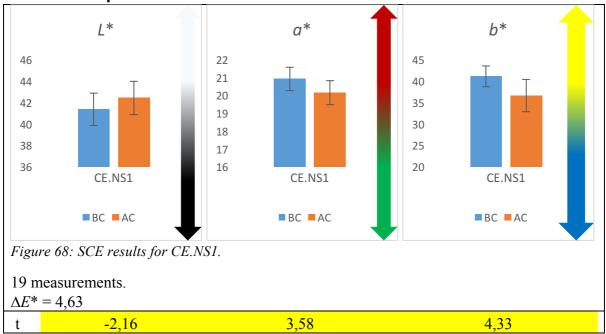


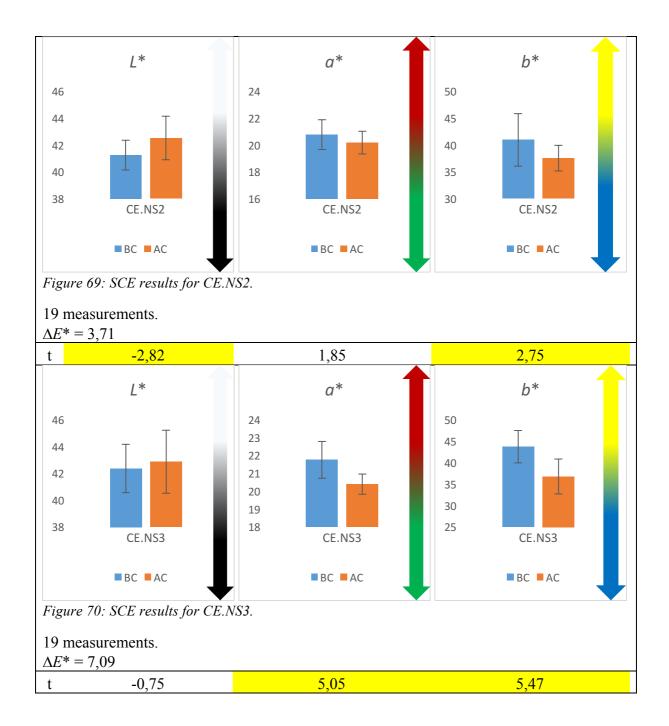


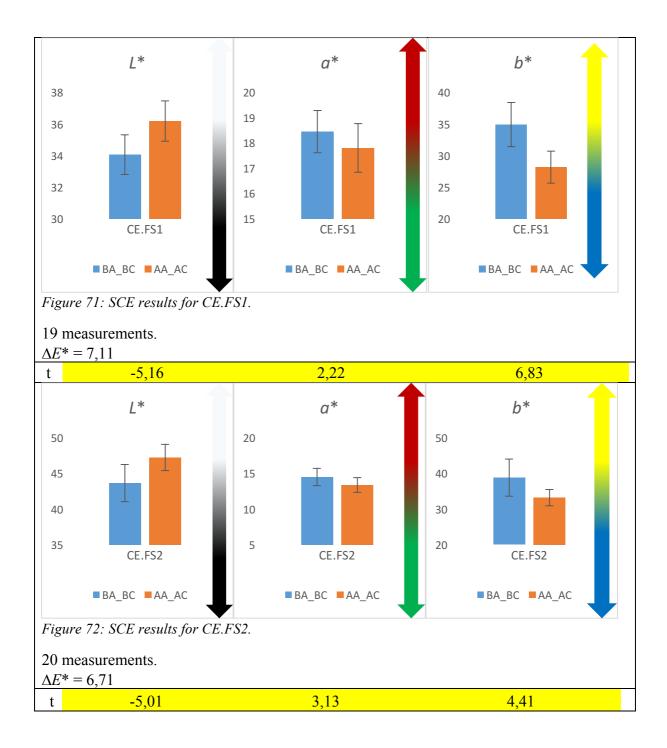


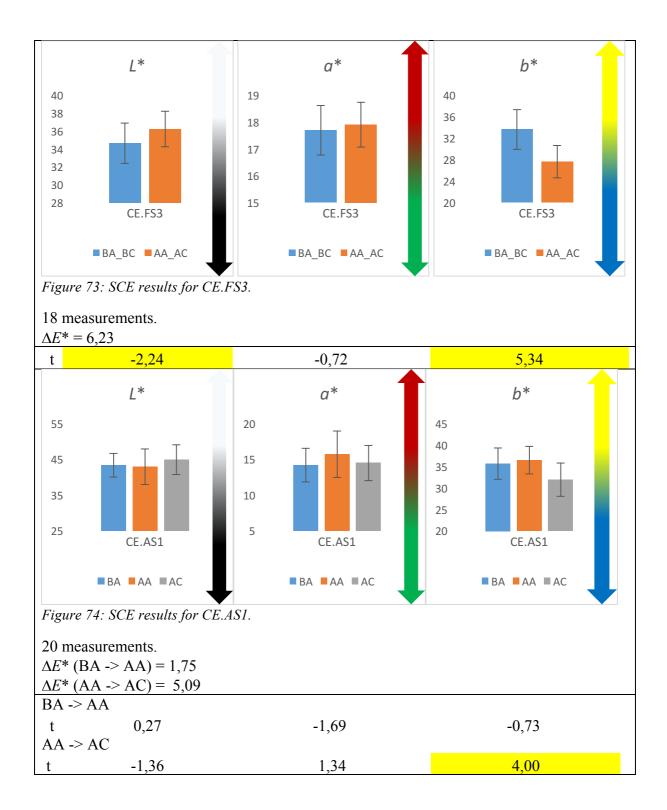


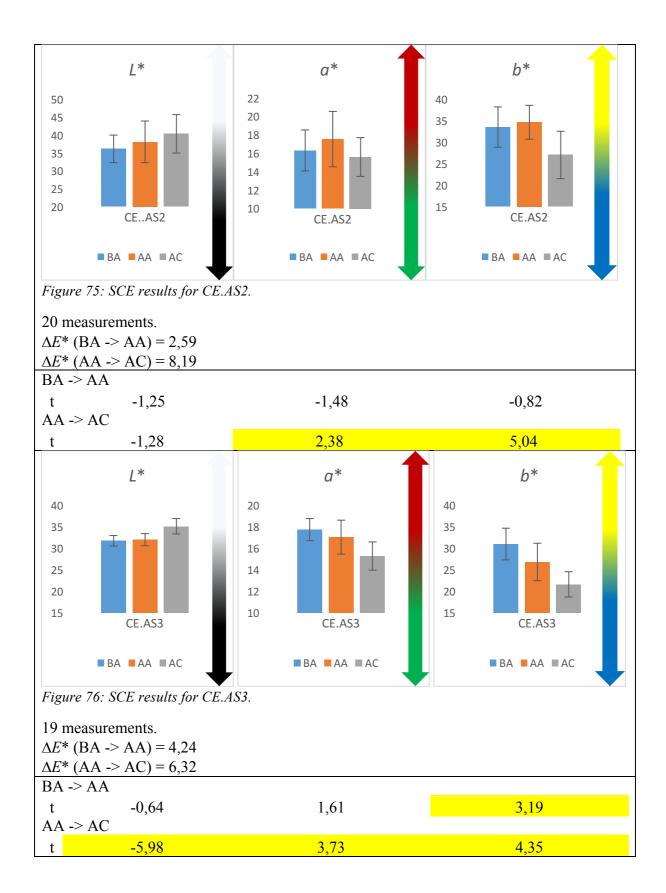
**Centurio samples** 



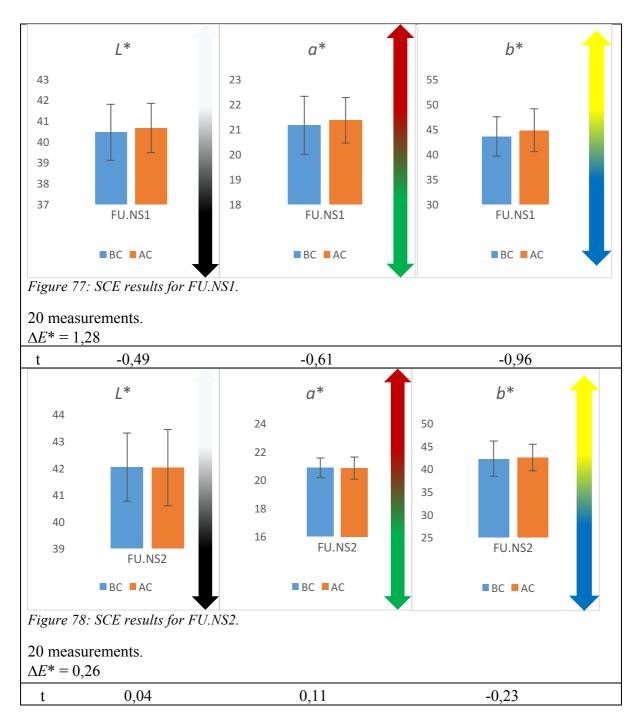


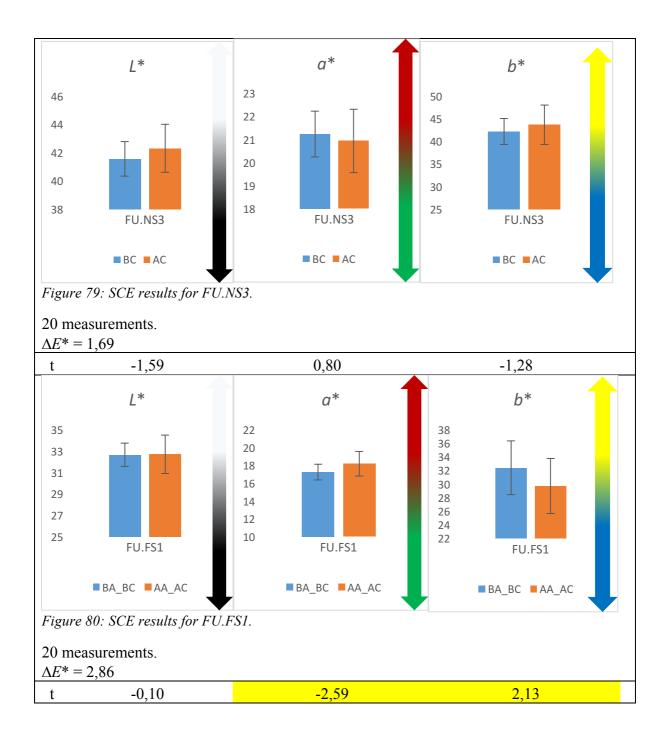


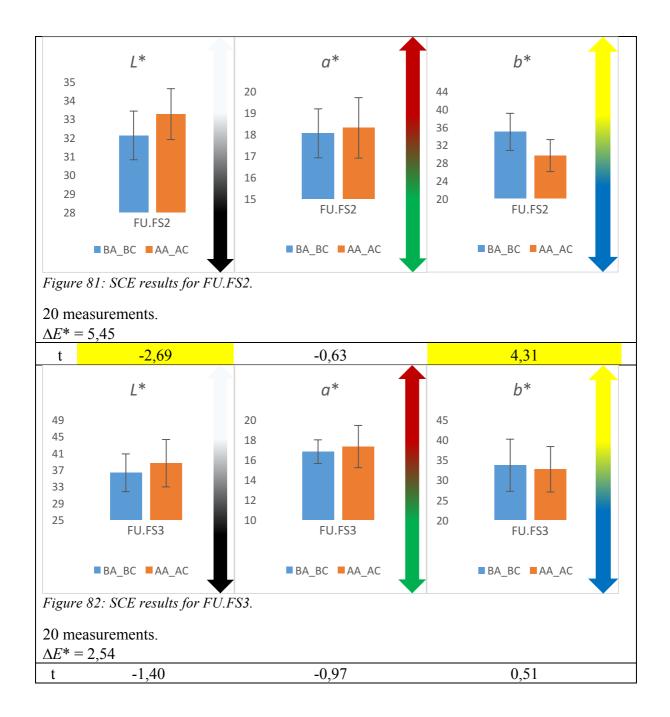


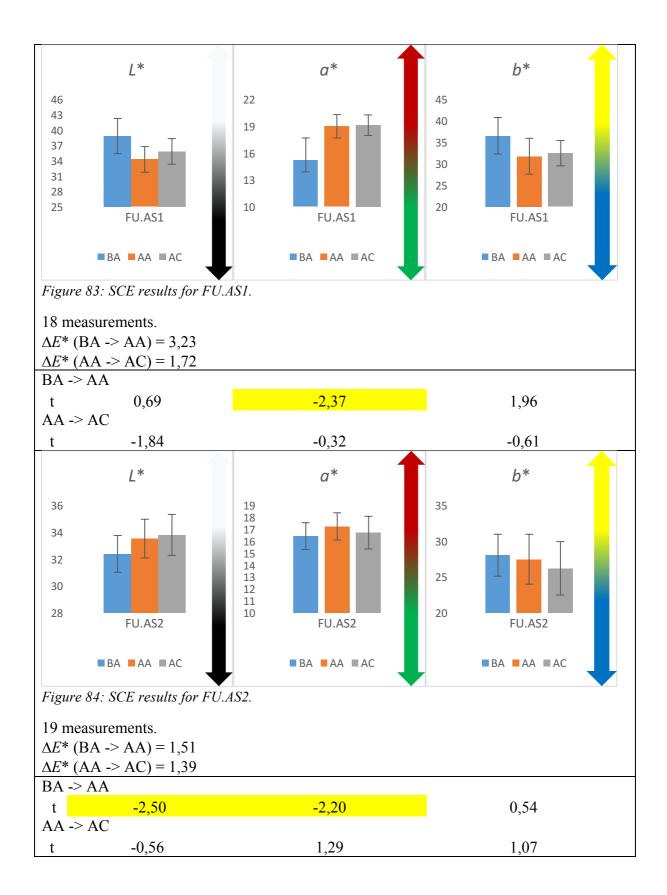


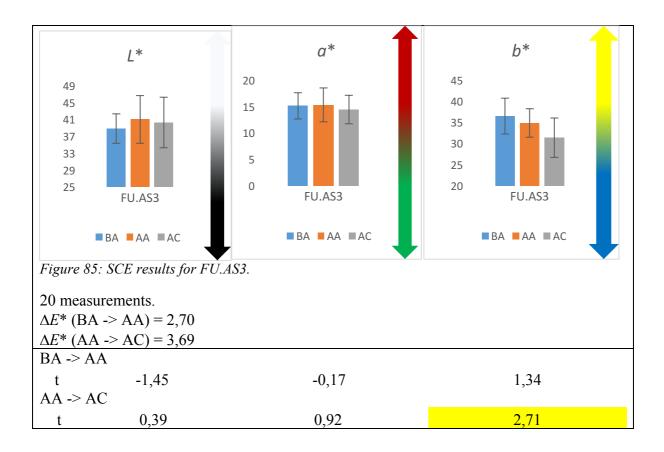
### **Fulgentin samples**

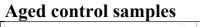


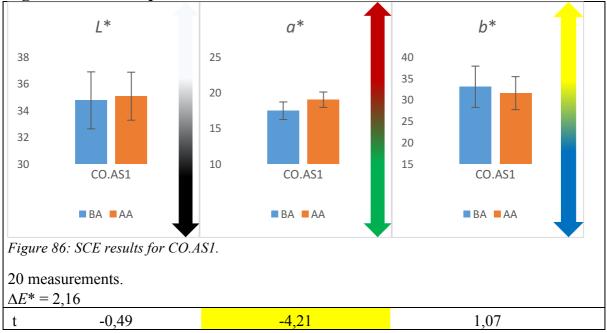


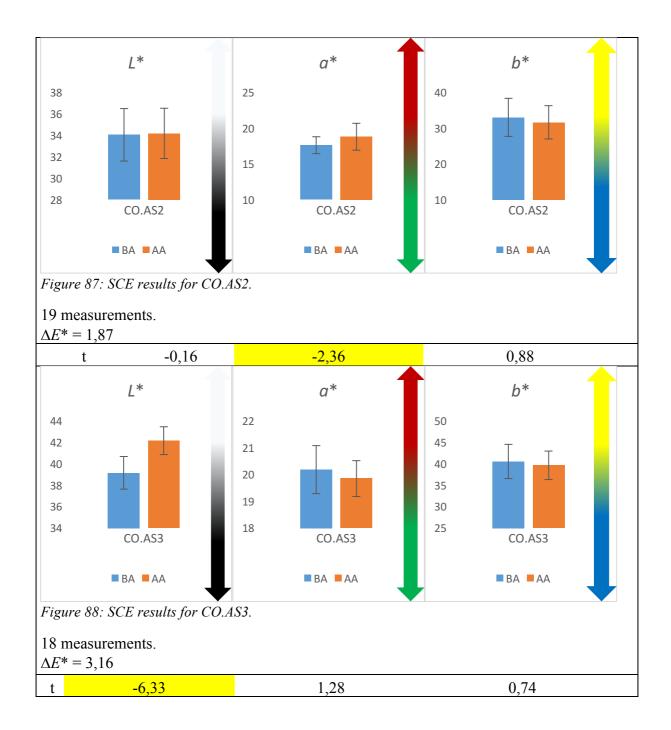




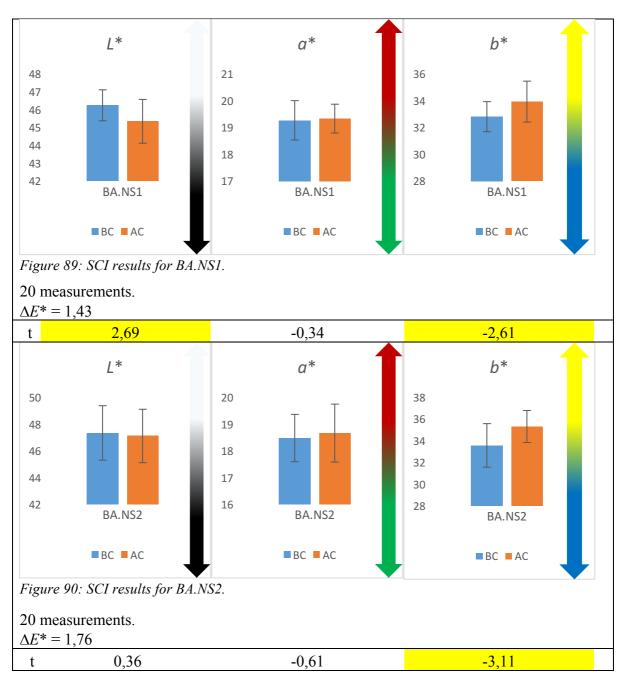


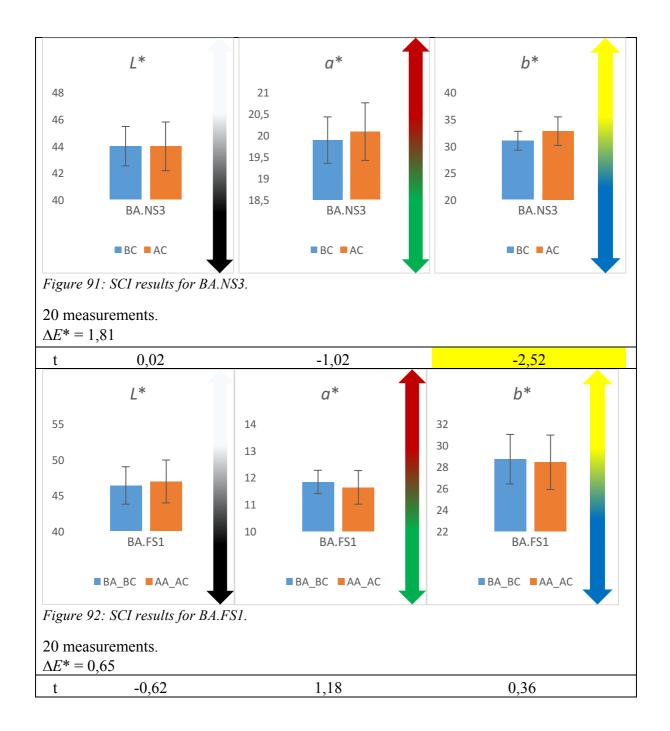


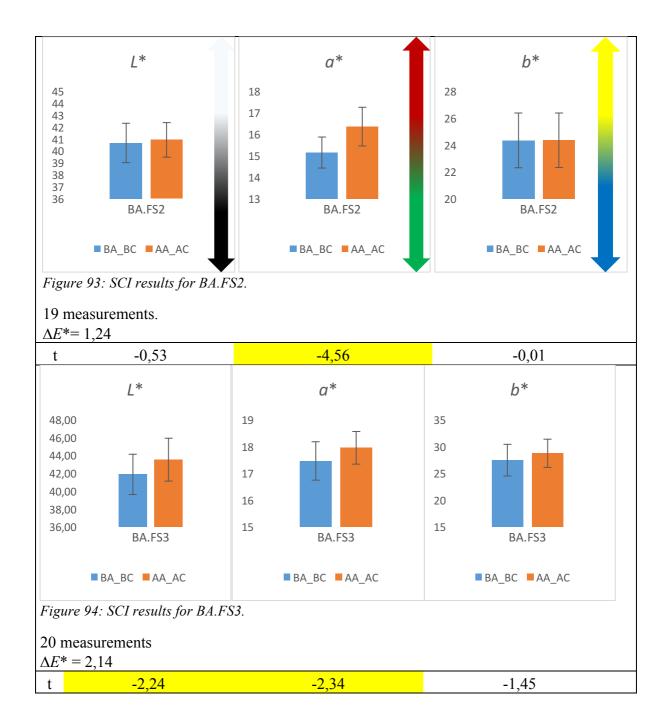


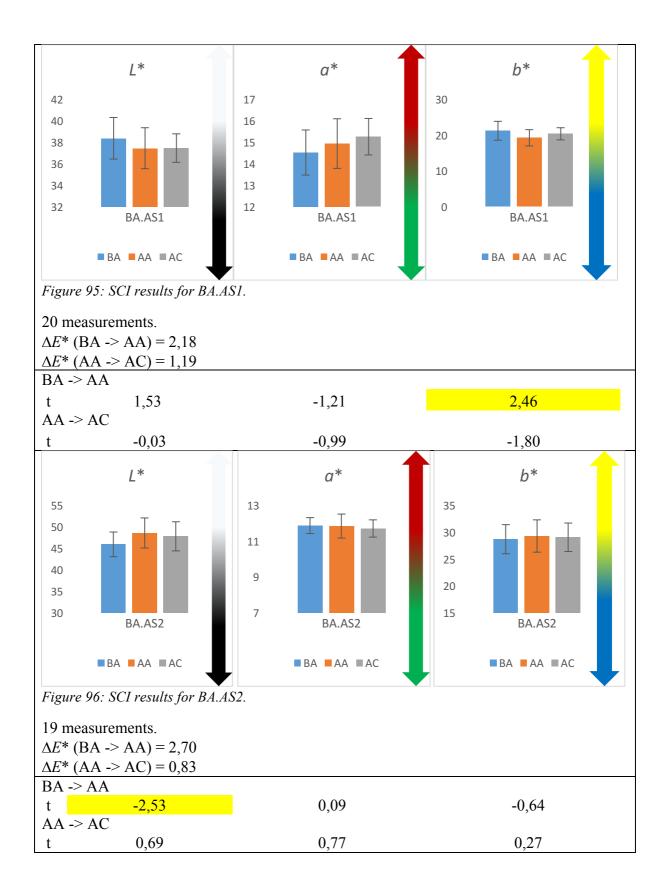


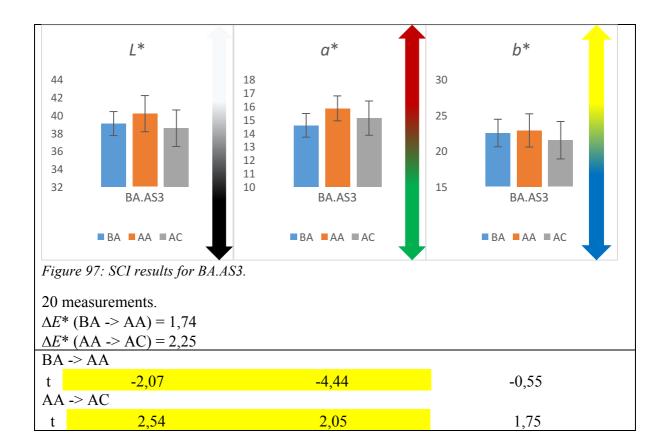
### SCI results Baolin results

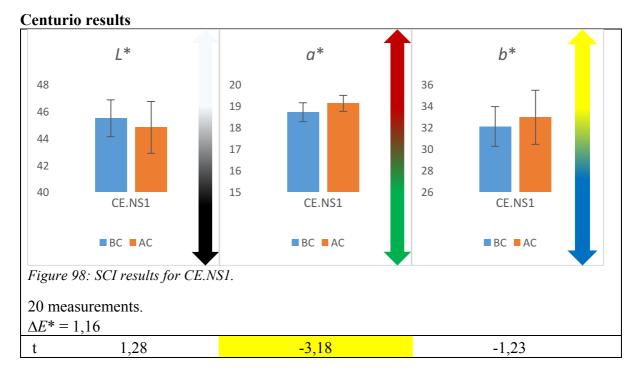


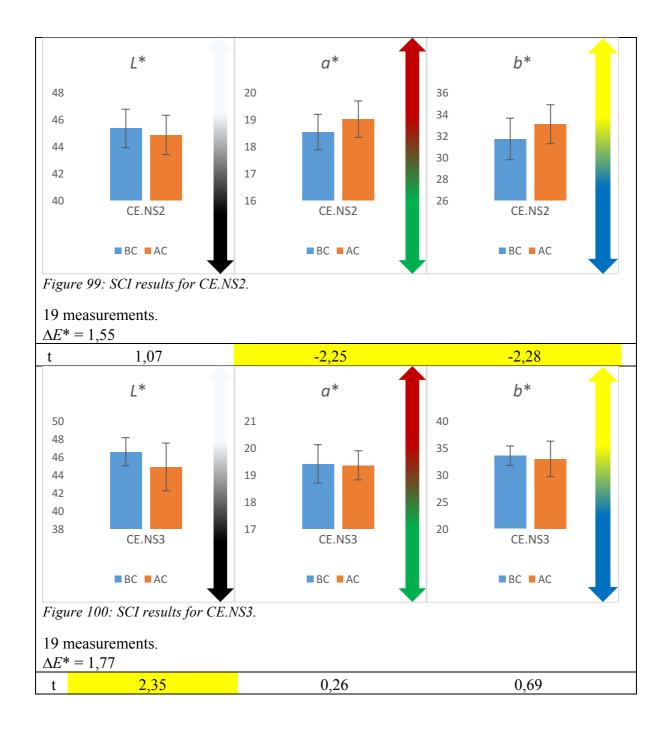


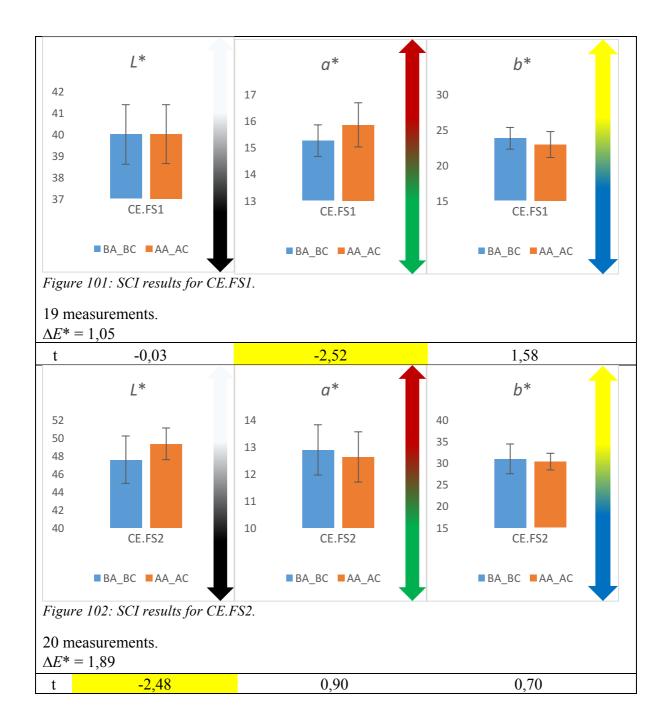


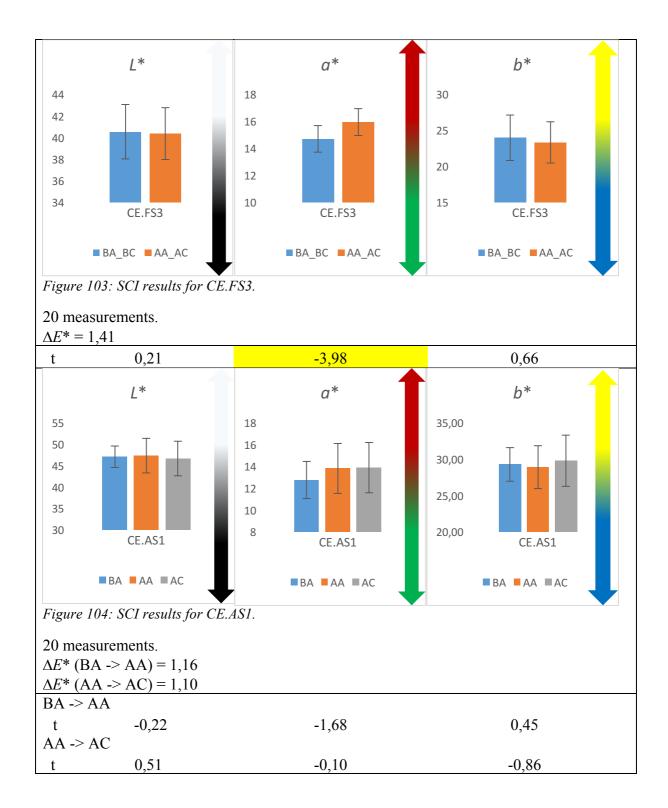


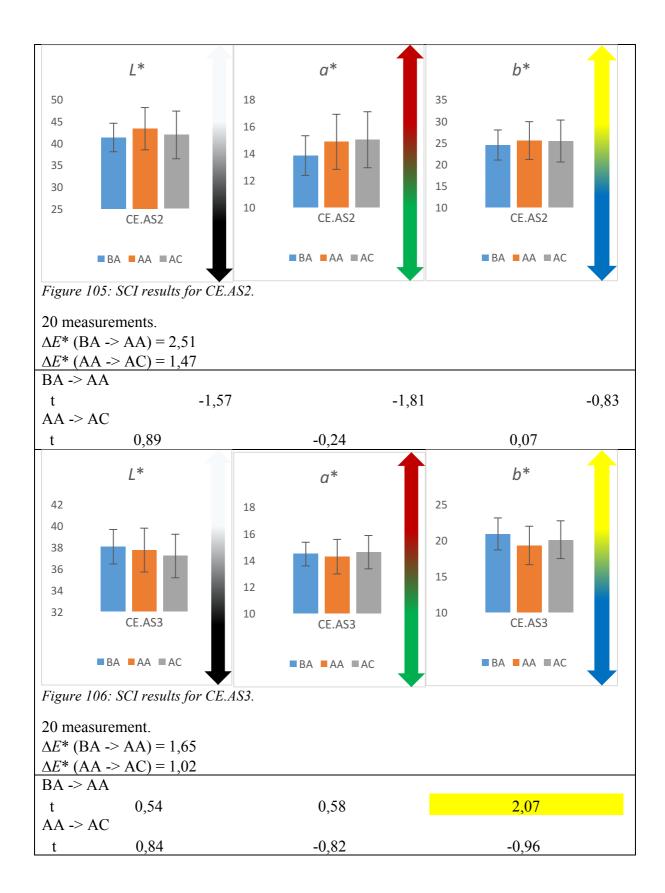




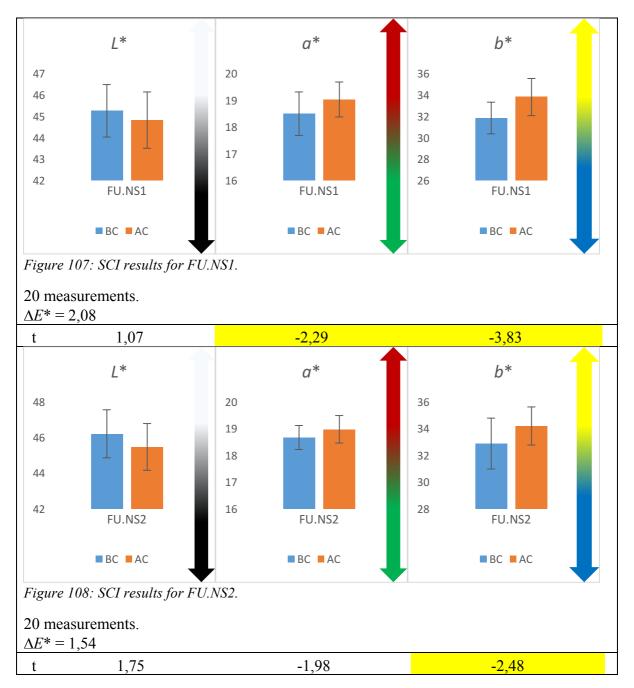


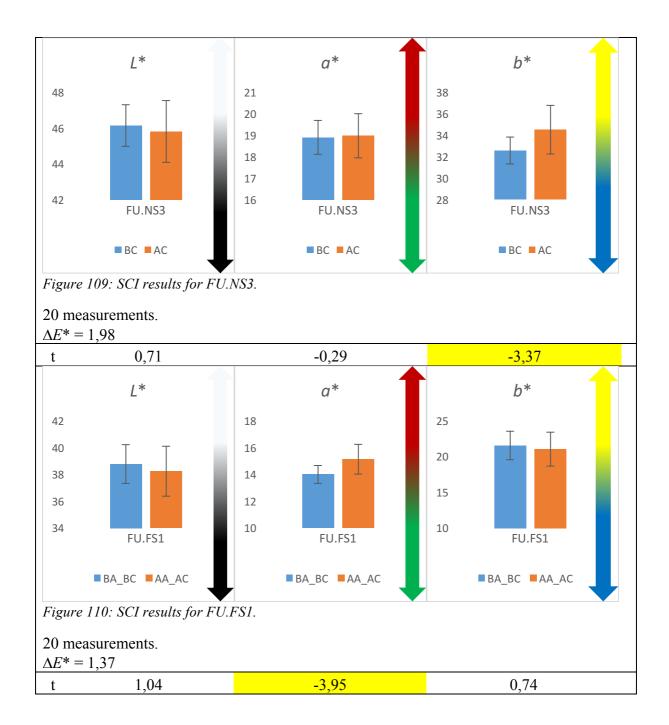


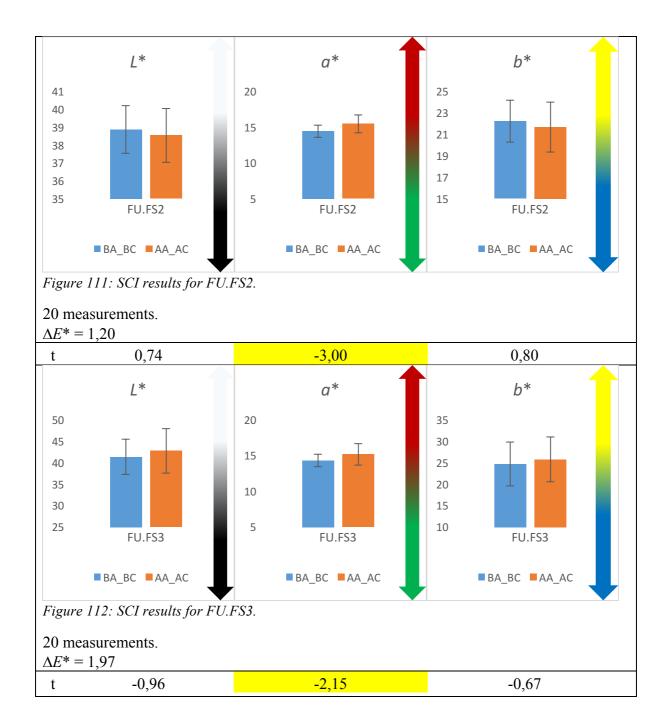


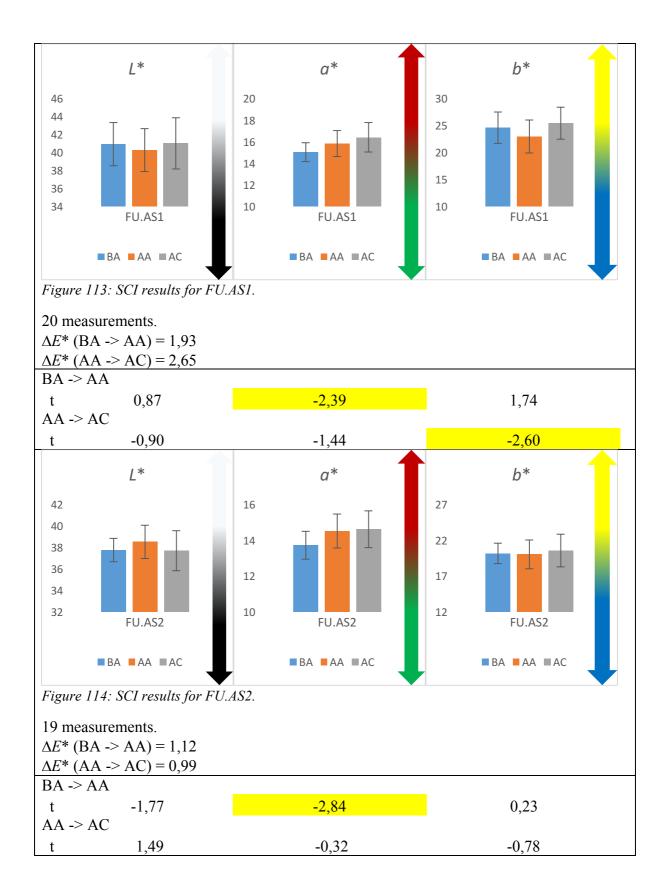


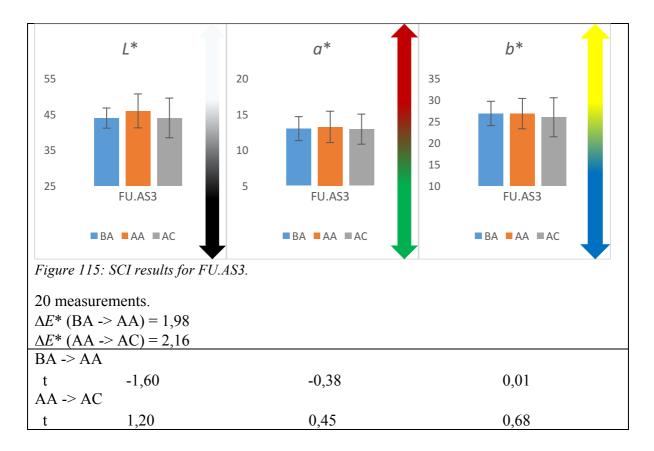
#### **Fulgentin samples**



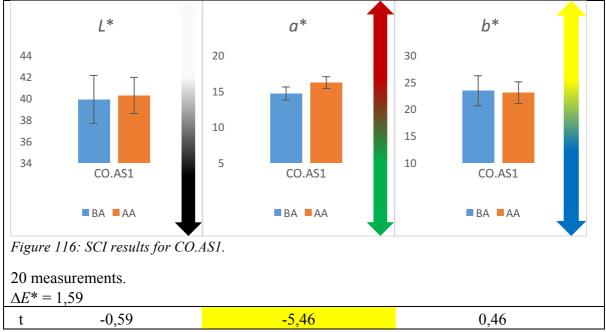


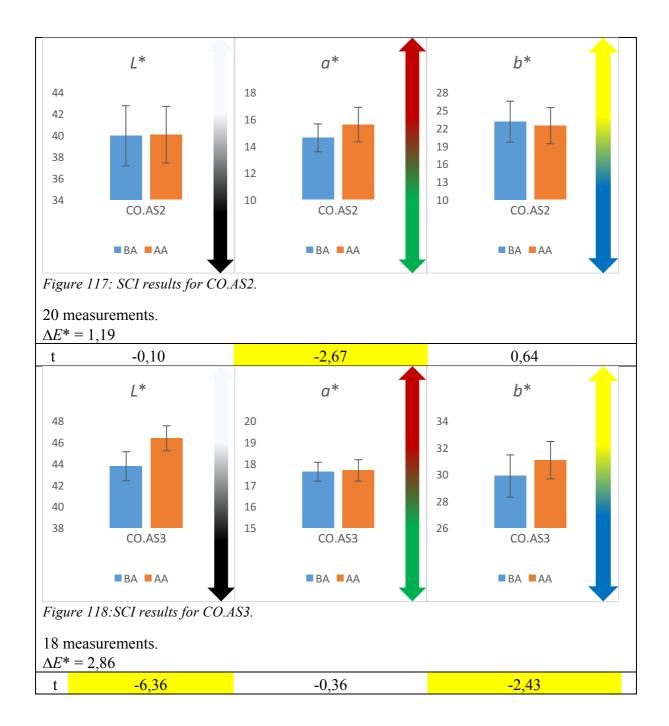






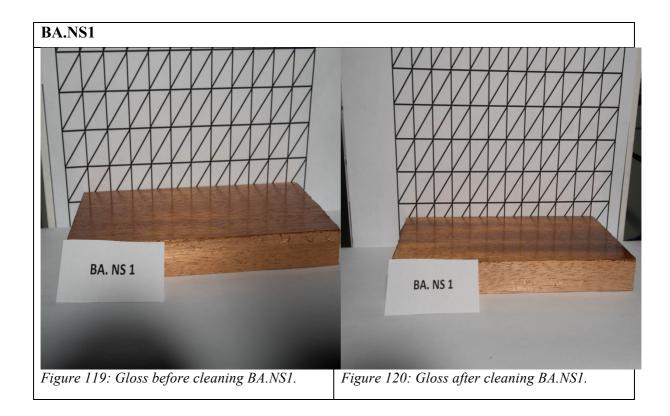
### Aged control samples

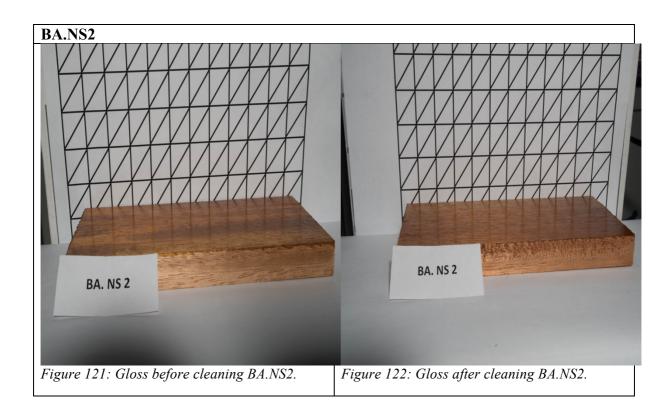


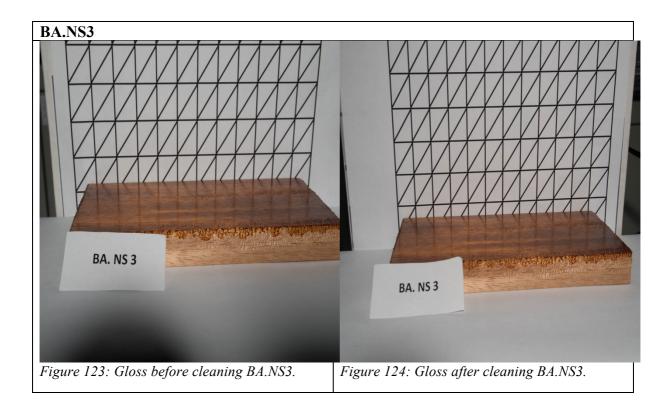


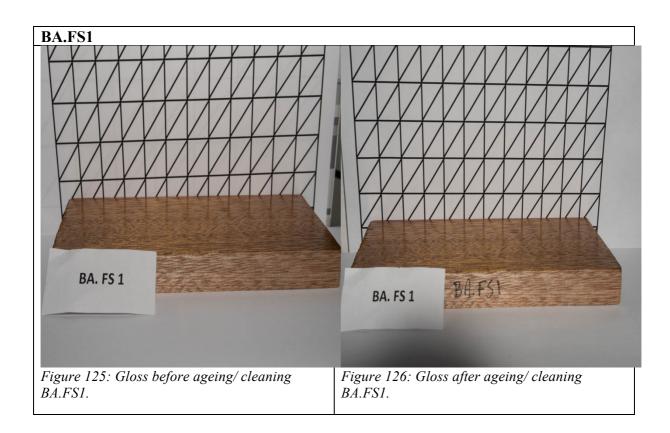
# 6.5 Appendix 5: Pictures of gloss measurements

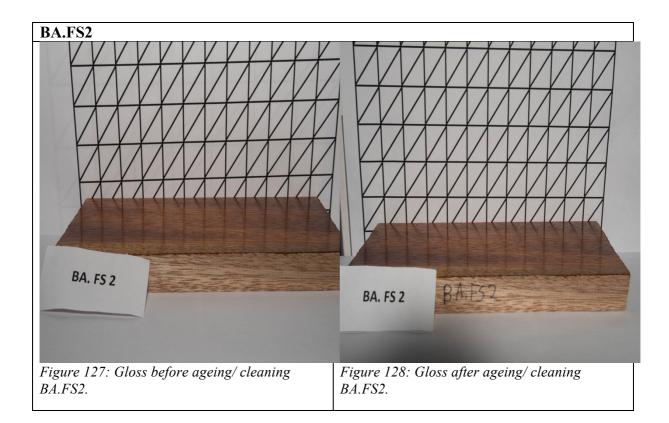
### **Baolin samples**

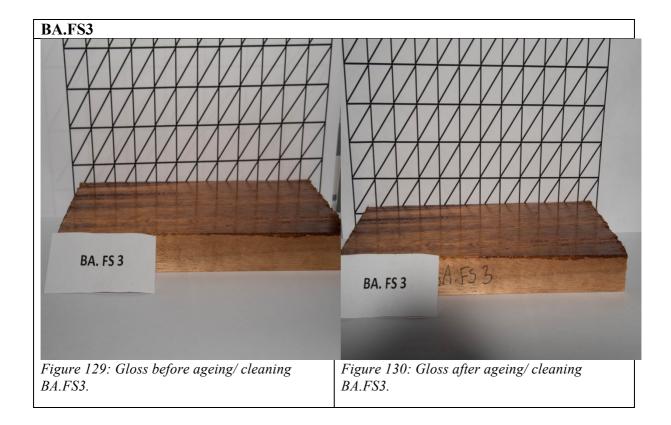


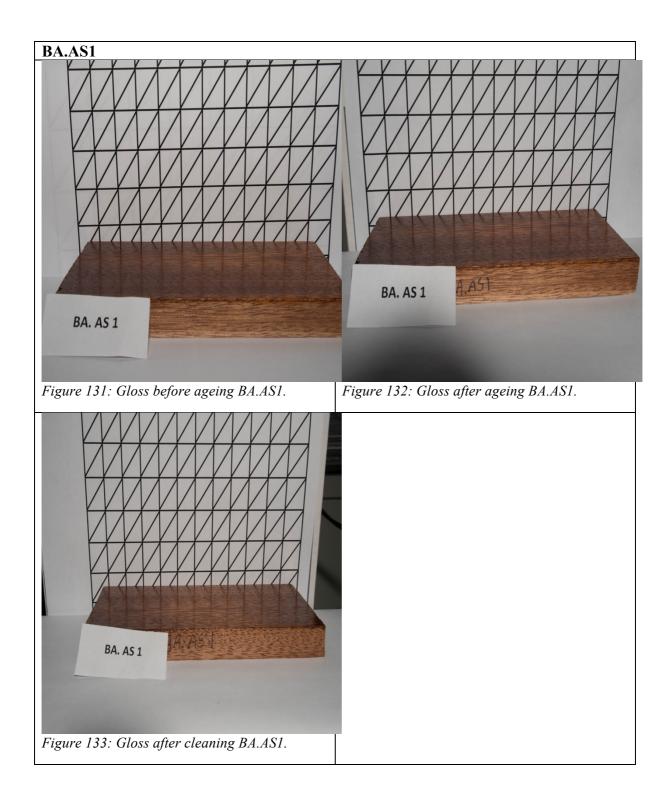


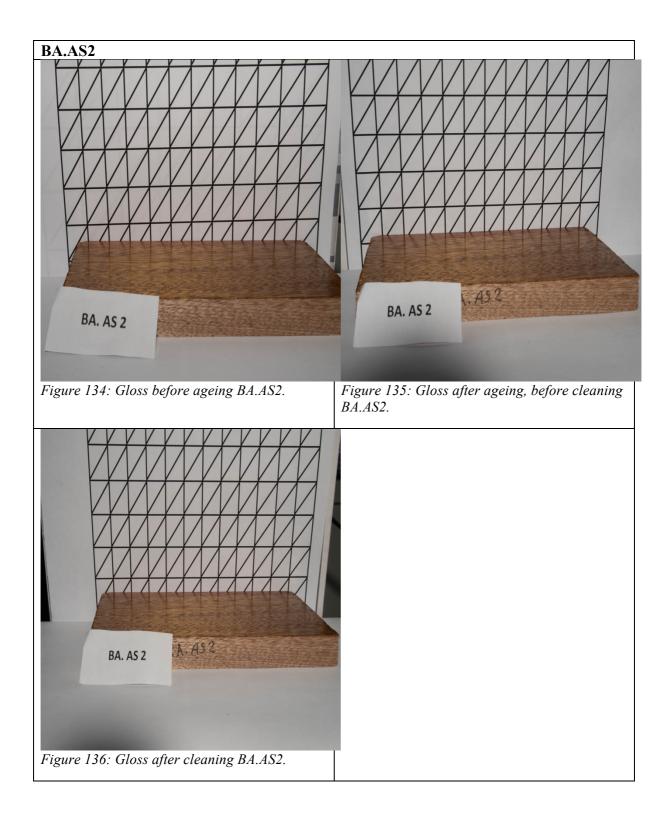


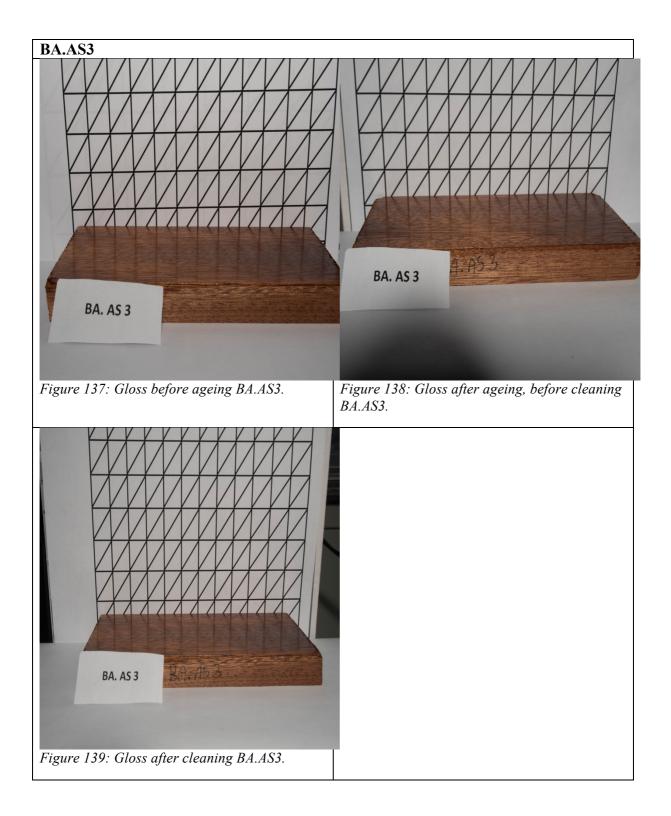




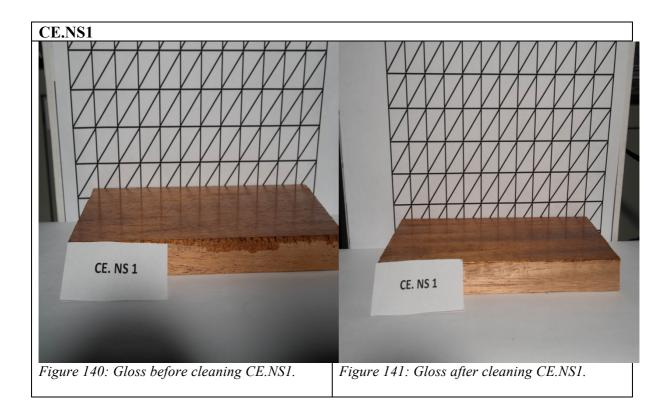


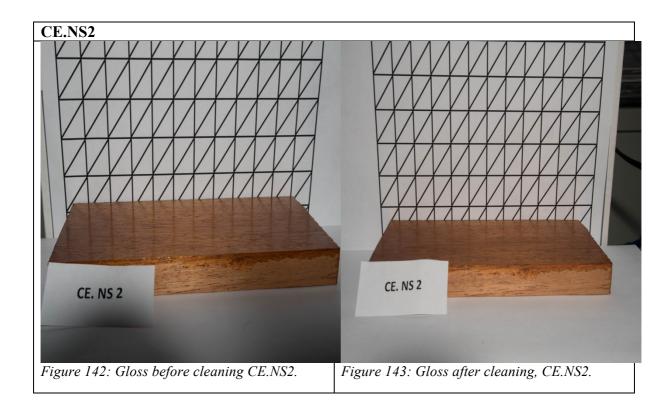


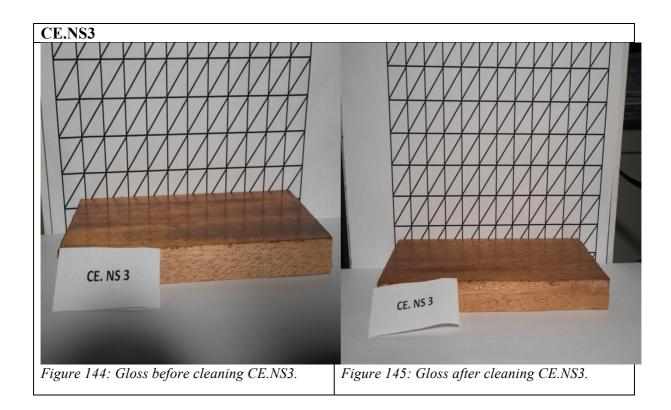


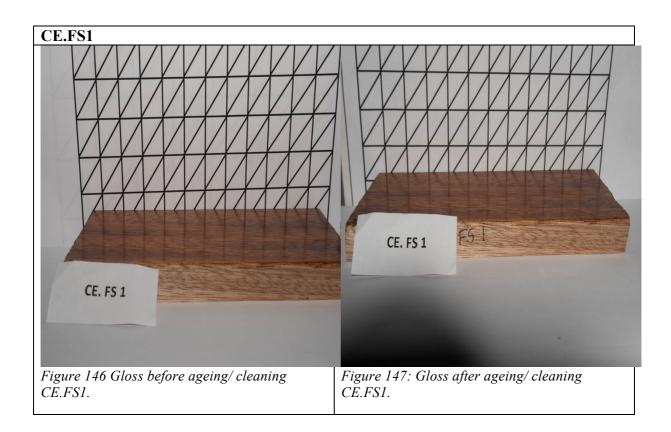


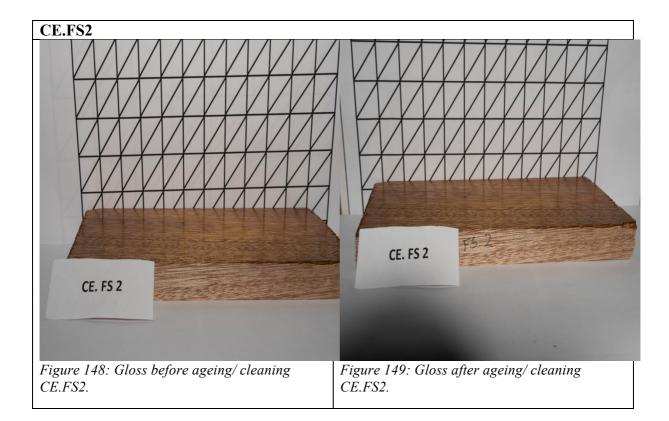
# **Centurio** samples

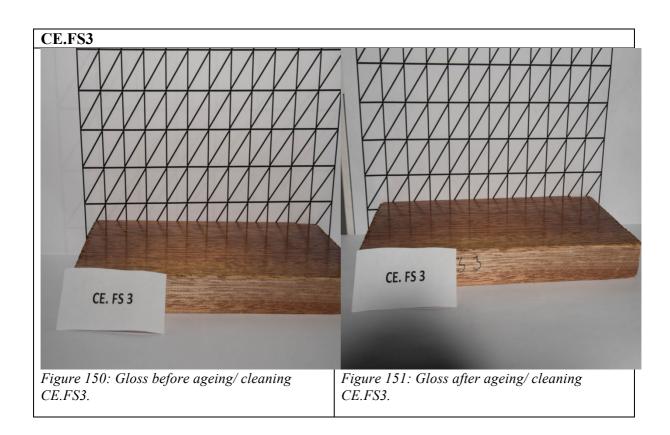


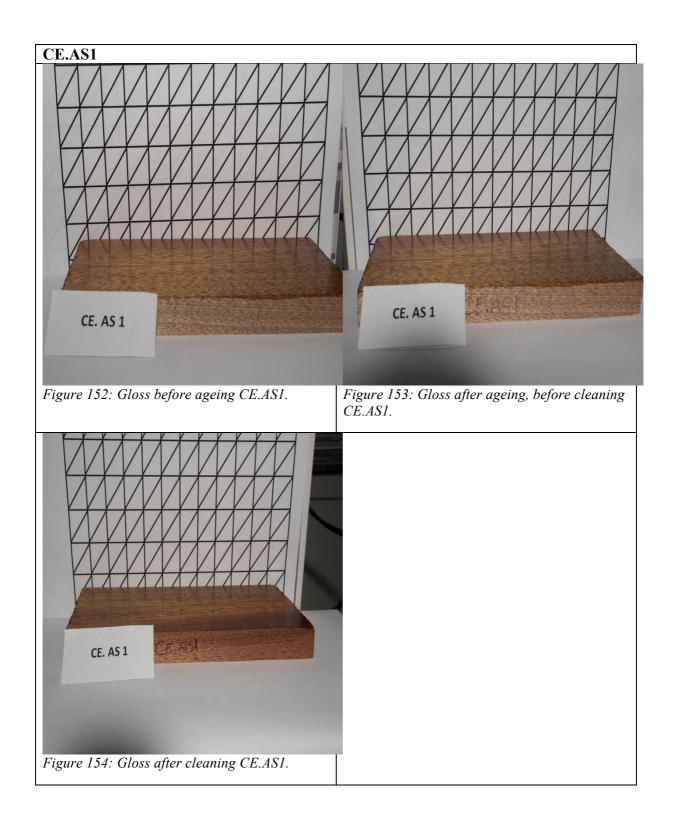


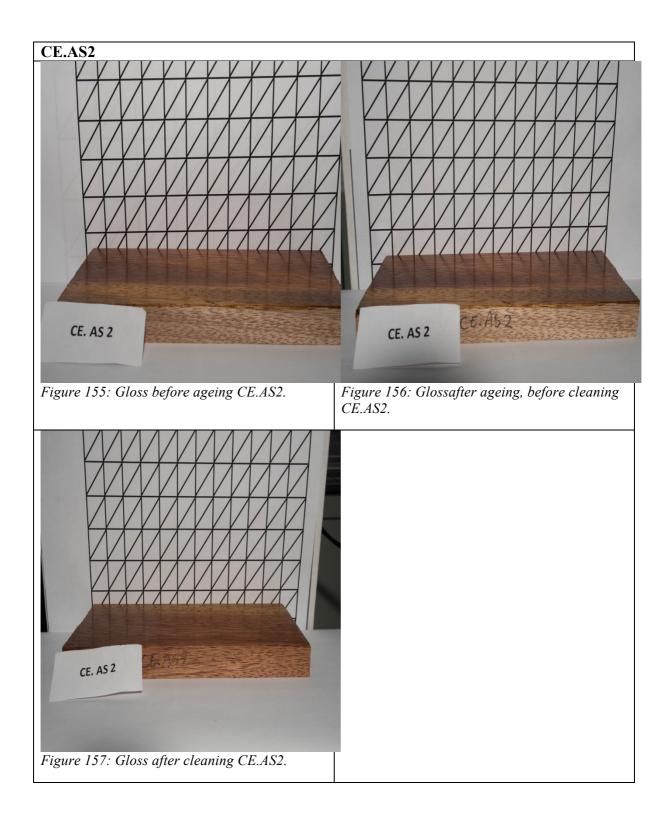


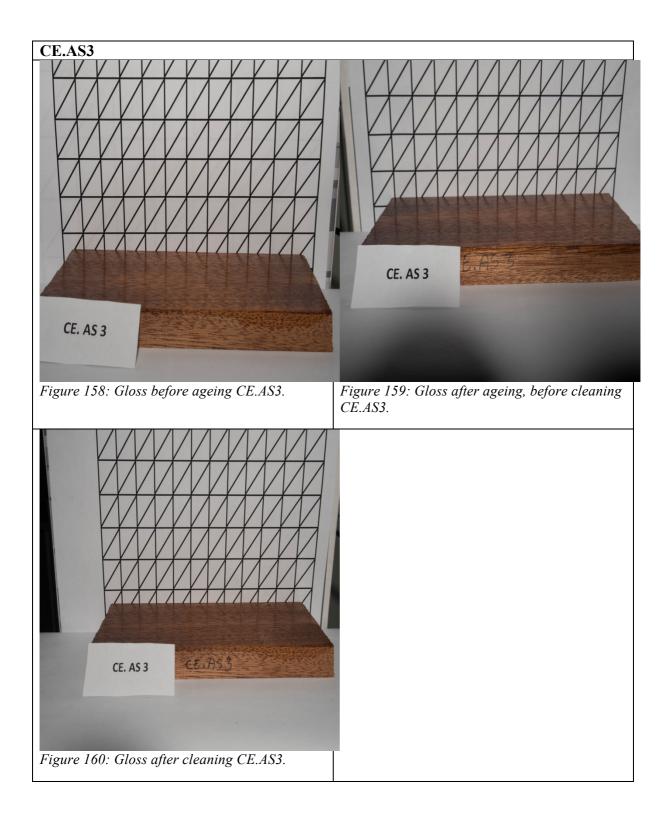




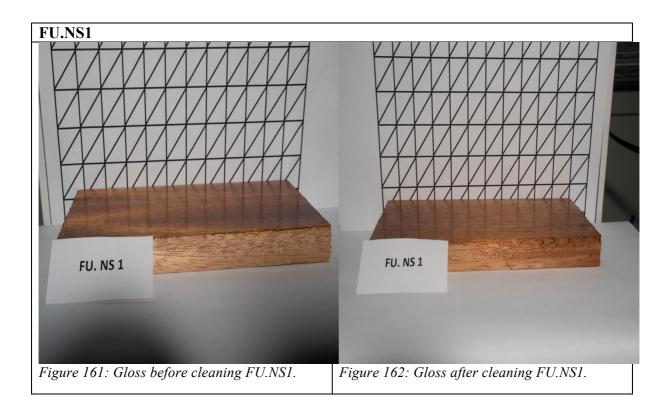


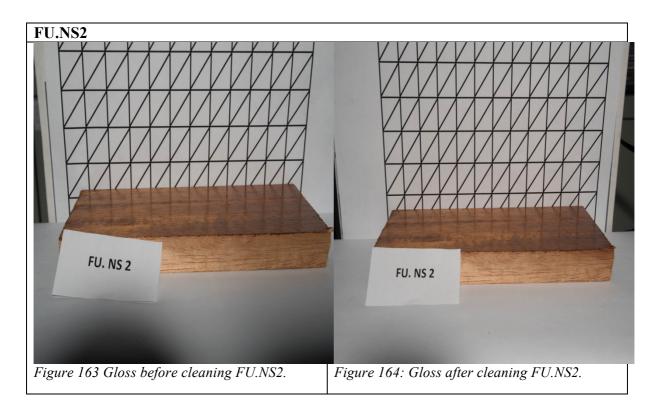


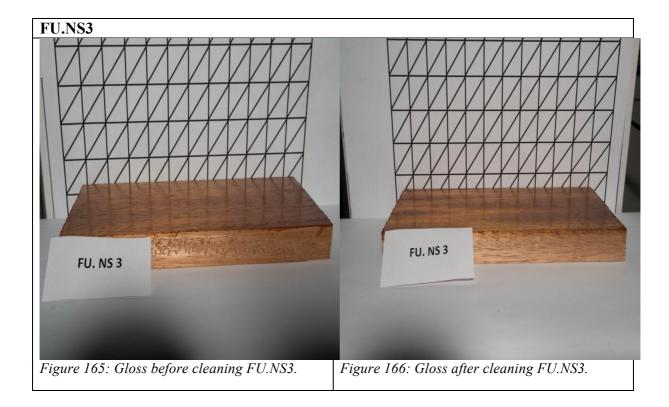


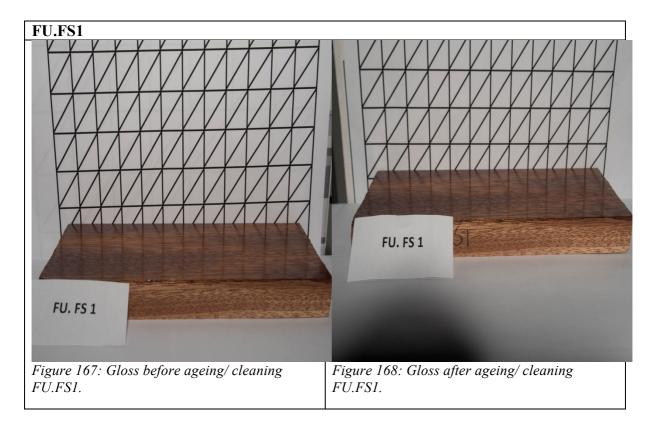


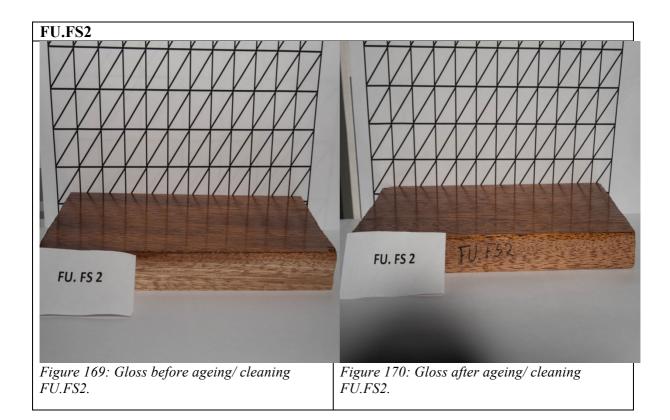
### **Fulgentin samples**

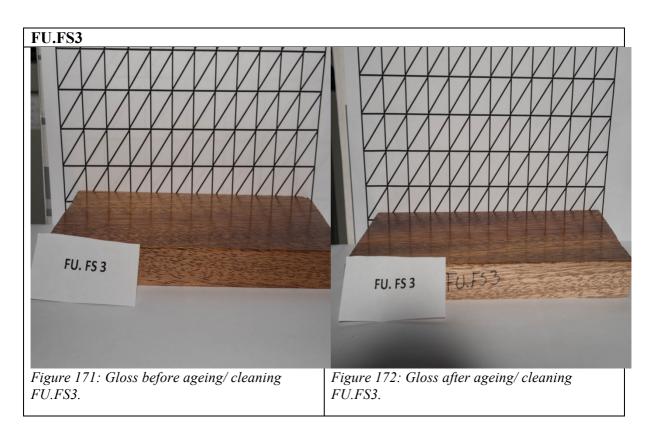


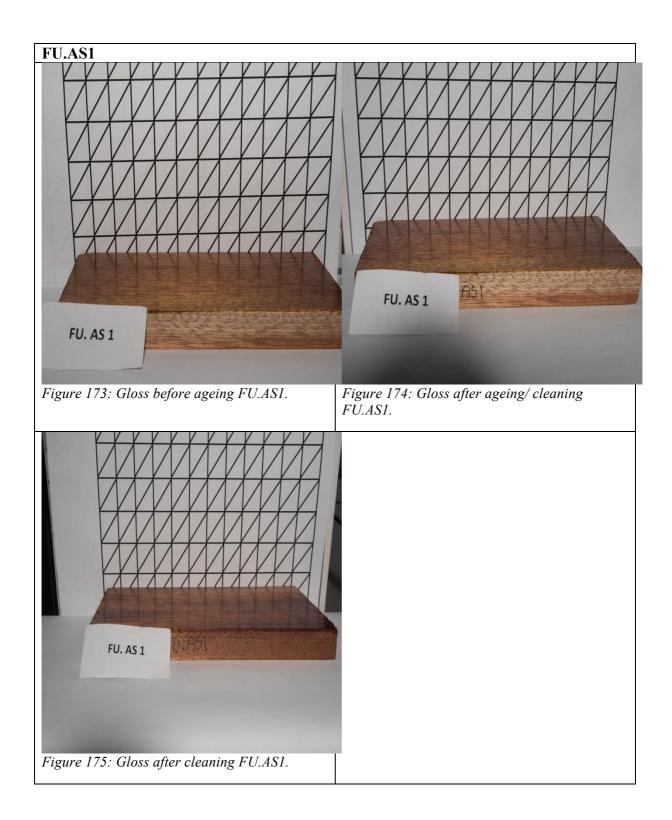


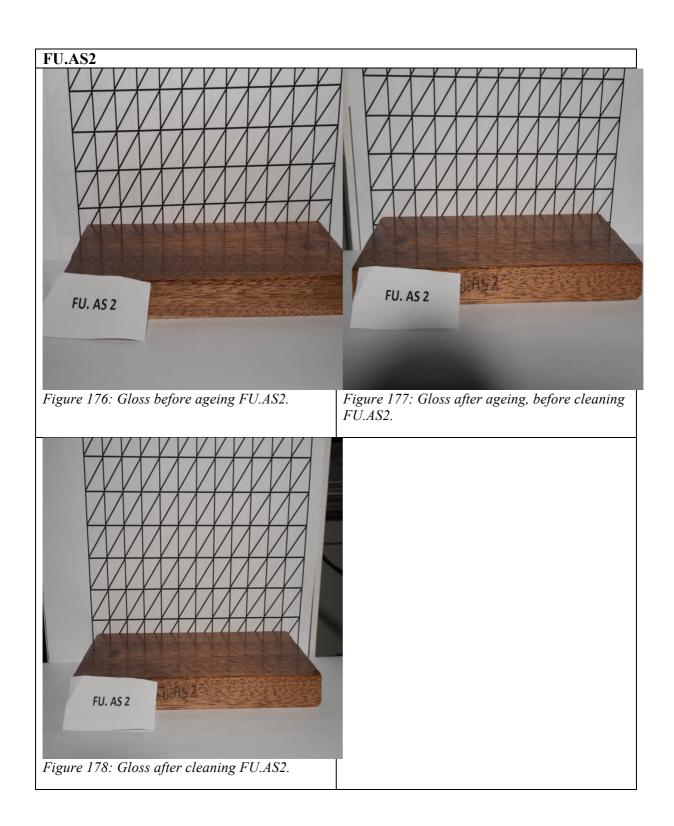


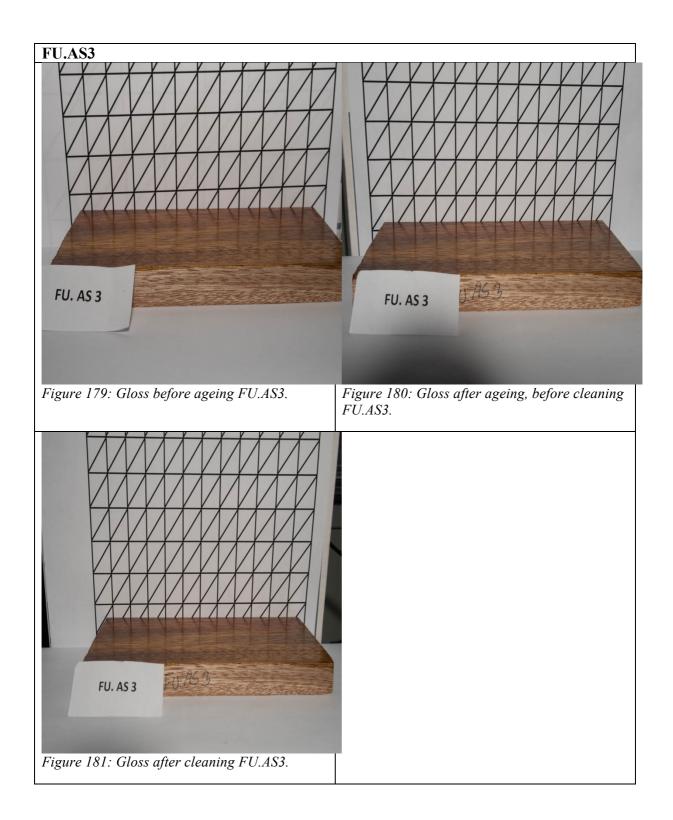




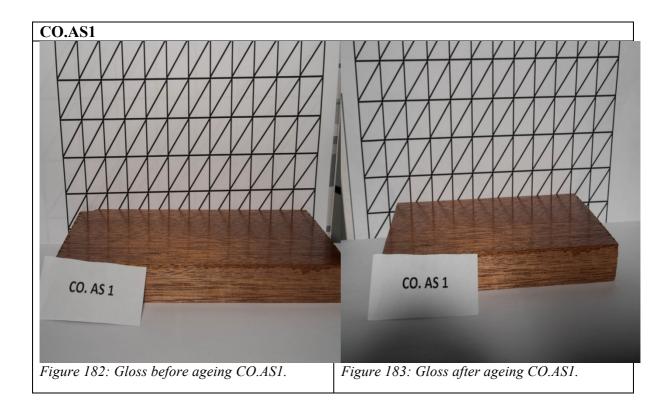


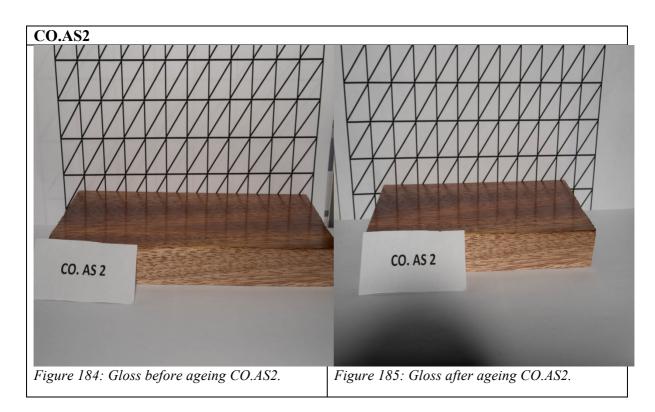


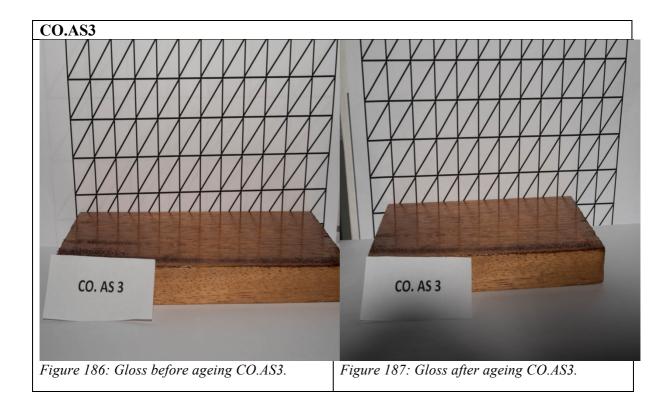




## **Control samples**







# 6.6 Appendix 6: Pictures of samples

# **Baolin samples**

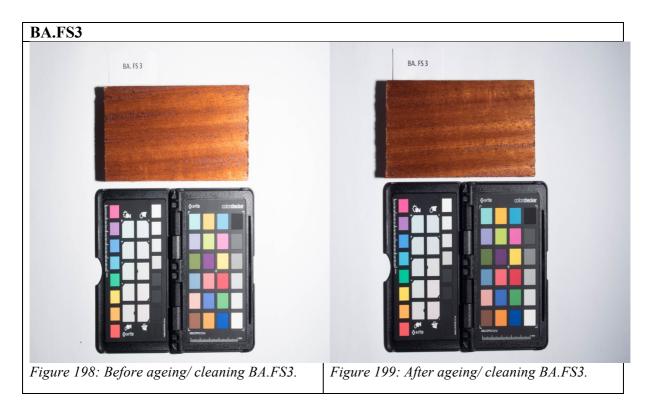




BA.NS3	
BA. NS 3	BA. NS 3
Figure 192: Before cleaning BA.NS3.	Figure 193: After cleaning BA.NS3.













## **Centurio samples**











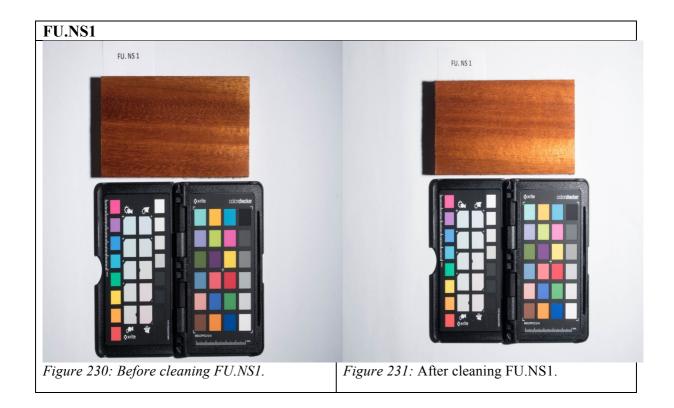








## **Fulgentin samples**

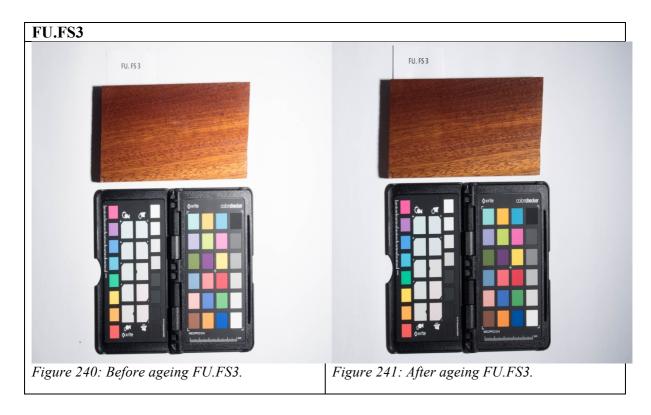


















## **Control samples**







