



## Evaluation of chia and flax mucilages as consolidants of paint films and as hydrogels used in the cleaning of canvases reverses: first results

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**Abstract** In the field of conservation of cultural heritage, animal glues, synthetic resins, and some polysaccharides are used to consolidate pictorial layers. Meanwhile, in order to clean the obverse and the reverse of paintings, organic solvents and aqueous systems are sometimes employed in the form of gels. In this work, vegetable mucilages have been tested for both applications. This research has been carried out in several phases. Firstly, mucilages were extracted from flax and chia seeds. The efficacy of the obtained products was tested on two kinds of mock-ups. As consolidantes, the mucilages were applied on a board with a film of powdery tempera painting. In the case of their use as cleaning gels, two linen cloths were prepared: one with animal glue and the other with *gacha*, an adhesive used in the Mediterranean Basin. In the last step, the efficacy and safety of the treatments were determined through microscopic observations and FTIR-ATR measurements. Colour and gloss characterisation were also performed in the consolidated pictorial layers. According to the first results shown in this work, flax mucilage can consolidate disintegrated layers of rabbit-skin glue tempera producing minimum colour and gloss changes, whereas chia mucilage has less efficacy to consolidate the tempera. Regarding the efficacy of mucilages as hydrogels, both have shown promising results. With these first results, future work will focus on the study of the long-term behaviour of vegetable mucilages through accelerated artificial ageing and on laying the foundations for their use in other conservation treatments, such as the cleaning of pictorial layers.

### 1 Introduction

The use of mucilages in the artistic field is not new. In the past, some pre-Columbian cultures already used certain mucilages in the creation of their art works before and during the Viceroyalty. One example of this kind of materials is *tzauhtli*, a mucilage obtained from the pseudobulbs of certain endemic orchids in Mexico. This product was used as an adhesive by *Mexican* artisans in the manufacture of feather mosaics, as a binder for certain pigments in mural paintings and in the paste to create some light sculptures, such as those popularly known as *Cristos de maíz* [1]. Over the years, they fell into disuse, at the beginning, due to the animal glues brought from Europe and, later on, to the appearance of synthetic adhesives [2].

In the field of conservation–restoration of cultural heritage, synthetic polymers and animal glues have been commonly used for consolidation or cleaning purposes. However, products such as *agar-agar* or *funori*, polysaccharides obtained from algae, which are not considered part of the plant kingdom, are finding gradually more use nowadays [3]. Nevertheless, only a few examples of the use of mucilages from vegetal origin as conservation materials can be found. The adhesive known as *tzauhtli* has been tested recently in Mexico as consolidant for highly degraded fabrics with satisfactory results [4]. Another example is the treatment applied to *Bandera Coronela del Batallón de Infantería Rey Fernando*, located in the Army Museum (Toledo, Spain), in which it was used a mixture of *tzauhtli*, *Methocel*<sup>TM</sup> (hydroxypropylmethylcellulose) and sorbitol, applied by impregnation with a brush (personal communication). On the other hand, the mucilage extracted from nopal, popularly known as *baba de nopal*, is used, especially in Mexico, as an additive in mortars in the field of building rehabilitation [5].

Mucilages can be found in different organs of the plants (bulbs, roots, stems, leaves, and flowers) and in seeds, especially in the outer tegument. They are heterogeneous polysaccharides constituted by macromolecules which consist of some monosaccharides, such as galactose, mannose, glucose and other glucid derivatives, linked by glycosidic bonds [6–8].

It is worth noting that there is some confusion between mucilages and vegetal gums since they are both polysaccharides with similar composition. Jean Bruneton recommends grouping all these plants products under the term hydrocolloids [6]. In any case, the simplest way to differentiate them is through their biological function: gums are substances exuded by plants in response to an

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external physical threat or other adverse conditions, so their function is to seal the injured area [9], whilst mucilages are part of the metabolism of the plant, the seed or the organism in general.

Several functions are attributed to mucilages, such as retaining water to prevent desiccation of plant tissues [8]. They also constitute a reservoir of nutrients for the plant, due to its polysaccharide nature. Mucilages also favour germination because they increase the contact surface between the seed and the earth substrate, increasing water diffusion [10, 11]. For their use as thickeners in the production of hydrogels, two properties are interesting: their ability to retain water [10], and their capacity to form highly viscous solutions at low concentrations, due to the presence of branched structures which contain hydroxyl groups that can form hydrogen bonds with water [11–13].

On the other hand, mucilages can be used in the elaboration of water-based glues due to their adhesive properties [11, 12], which make them interesting as consolidants of disintegrated paint films.

In this work, the authors propose the application of some mucilages obtained from vegetal organisms, rather than synthetic resins, in the field of conservation and restoration of cultural heritage, in accordance with the current trend to use biodegradable materials whose processing does not imply a significant carbon footprint. Therefore, the aim of this research is to explore the possibilities of mucilages extracted from not expensive seeds, easily acquired in the market, like flax and chia, (as opposed to *baba de nopal* or *tzauhtli*, whose raw materials are more difficult to find in Spain), to obtain products that could be suitable to consolidate tempera pictorial layers and to make hydrogels to remove natural adhesives in the reverse of paintings. The study of these possibilities can determine if they could constitute new and good alternatives to the products currently used for these purposes.

## 2 Materials and methods

The methodology applied in this work can be summarised in four steps:

- 1- Extraction of the mucilages from the seeds.
- 2- Characterisation of the obtained products.
- 3- Preparation of mock-ups to test the mucilages as consolidants of pigments in tempera paintings and as hydrogels to eliminate natural adhesives.

Three mock-ups, imitating different artistic objects, were prepared. One of them (Mock-up A) was made to simulate a film of rabbit glue tempera painting with a deficiency in the binder that was applied on panel. The objective for this mock-up was testing the effectiveness of chia and linseed mucilages as consolidantes to restore the cohesion of glue tempera pictorial layers, given the adhesive capacity of mucilages.

The other two mock-ups were made imitating canvas paintings reverses, to which two different types of natural adhesives, widely used in the Mediterranean area, were applied: rabbit skin glue (Mock-up BI) and *gacha* (Mock-up BII). In this case, mucilages were used as gels due to their ability for absorbing water, to test their capacity for eliminating such adhesives.

In both cases, the aim was to make a first approximation of the necessary methodology to check their effectiveness, in the field of conservation of cultural heritage, making an incipient assessment of the method of application, in the case of consolidants.

- 4- To study the mucilage's efficacy for such treatments and their safety by analysing the possible changes in colour and gloss of the surfaces, as well as the presence of rests of *gacha*, rabbit skin glue and mucilages after the treatments.

To prepare the mock-ups, the materials summarised in Table 1 were used:

### 2.1 Equipment

To characterise the extracted mucilages and to analyse the cleaned surfaces, Fourier transform infrared spectroscopy (FTIR) with Attenuated Total Reflection (ATR) has been used with a Fourier transform infrared spectrometer (*Thermo Scientific Nicolet 380*) with a DTGS (deuterated triglycine sulphate) temperature-stabilized coated detector and equipped with an attenuated total reflection

**Table 1** Materials used in the elaboration of mock-ups and commercial brand in brackets

	Mock-up	
	Consolidation: Mock-up A	Aqueous cleaning gel: Mock-up BI Mock-up BII
Support	Plywood board (30 × 24 cm)	Two Velázquez type linen canvases placed on 22 × 27 cm stretchers
Ground	Ground made with rabbit-skin glue ( <i>CTS</i> ) and plaster of Paris ( <i>M. Barrero</i> )	Mock-up BI: rabbit-skin glue ( <i>CTS</i> ). Mock-up BII: <i>gacha</i> *
Pictorial layer	Tempera paint, made with rabbit-skin glue ( <i>CTS</i> ) and ultramarine blue pigment ( <i>Sennelier</i> )	—

\**Gacha* is the most common adhesive used in the Mediterranean countries to line canvas paintings. It has been prepared from *colletta*, wheat flour (*Gallo*), water and Venetian turpentine (*Talens*). *Colletta* was made of animal glue (*CTS*), water and vinegar (*Alipende*)

**Table 2** Mucilage extraction procedure

Extraction procedure		
Raw material	Chia seeds	Flax seeds
Origin	Brand <i>San Blas</i> . Chile	Brand <i>San Blas</i> . Poland
Proportion of seed: water (w/w)	1:50	1:40
Extraction	The mixture of seeds and water was heated at 70 °C during one hour with constant stirring at 1200 rpm. Then, it was introduced in an ultrasound bath for 10 min to accelerate the separation of the mucilage. After that, the mixture was filtered. The solutions were heated at 80 °C with constant agitation, at 1200 rpm, until it reduced its volume to half [16, 17]	
Purification	The solutions were heated at 55°C, and ethanol was added in a 1:1 volume ratio. The mixtures were shaken vigorously for 10 s and allowed to stand for approximately 20 min, whilst the mucilages began to coagulate. They were then separated by filtration [18]	
Drying	Mucilages were dried in a stove at 42°C for 24 h and then, milled using a glass mortar	
Final product appearance	Brown and shiny powder	Golden and shiny powder

diamond crystal accessory. The spectra were obtained in absorbance mode from 60 scans, in the range 4000–400  $\text{cm}^{-1}$  at 4  $\text{cm}^{-1}$  resolution and were analysed using Omnic v 7.3 and processed with Origin v 7.0.

To study the possible colours and gloss changes occurred after the consolidation treatment, the prepared mock-ups were studied, checking various areas before and after the application. For the colour, it was used a *CM-2600d* spectrophotometer from Konica-Minolta, in a wavelength range of 400–700 nm and at an interval of 10 nm. This equipment works with the standard illuminant D65, standard 10° observer, reflection optical geometry (d/8) and measurement area diameter of 3 mm. The data were acquired by using the CM-S100w 1.91.0002 SpectraMagic software and processed in a spreadsheet. Three colour measurements were made in each area, subsequently calculating their average value. To determine the colour variations experienced, it was used the CIE2000 formula [14]. Possible gloss changes were verified by using a TRIO NK-Glossmeter from Neurtek, making four measures in each area, choosing the 85° angular gloss.

To verify the effectiveness of the mucilages in the elimination of natural adhesives from the mock-ups BI and BII, and the possible rests after the treatment, the surfaces were observed, firstly, using natural direct and raking light and, subsequently, with a digital video microscope MOD.AM4113 T-FVW from Dino-Lite, using both visible and UV lights.

### 3 Experimental phase

#### 3.1 Extraction of mucilages

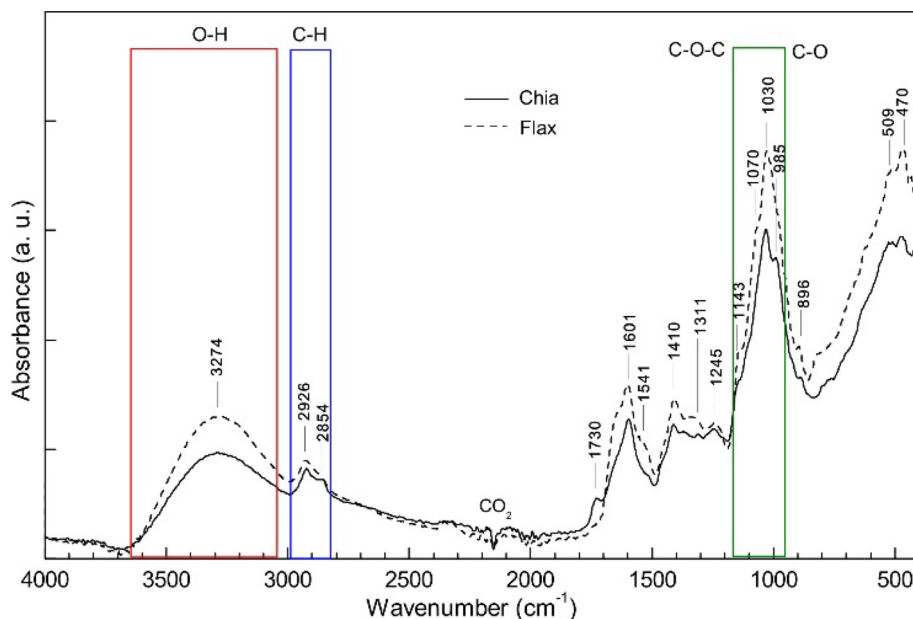
Some aspects were considered in the selection of the mucilages, such as their commercial availability and the easy reproducibility of the extraction process.

To obtain mucilages from flax and chia seeds, it has been used an aqueous process in all cases, employing deionised water, following the method indicated in Table 2.

#### 3.2 Characterisation of mucilages

To characterise the products obtained, the samples were subjected to FTIR analyses. The spectra obtained are shown in Fig. 1. They show the characteristic bands of a polysaccharide: the bands in the 950–1150  $\text{cm}^{-1}$  range are attributed to glycosidic (C–O–C) bonds [17]. In this range, bands corresponding to arabinose at 1143  $\text{cm}^{-1}$ , mannose around 1070  $\text{cm}^{-1}$  and glucose at 1030  $\text{cm}^{-1}$  are also found [19, 20]. The broad band around 3274  $\text{cm}^{-1}$  can be ascribed to O–H stretching vibrations [20]. Symmetrical C–OO stretching causes bands around 1410  $\text{cm}^{-1}$ , and the absorption at 1601  $\text{cm}^{-1}$  is the result of asymmetric C–OO stretching. In the chia mucilage, small bands appear at 1730  $\text{cm}^{-1}$  and 1541  $\text{cm}^{-1}$ , which can be attributed to the carboxylate group of uronic acid [21, 22]. Bands at 2926 and 2854  $\text{cm}^{-1}$  refer to C–H absorptions; these include CH, CH<sub>2</sub> and CH<sub>3</sub> stretching and bending vibrations, both symmetric and asymmetric, and occasionally double O–H overlaps [17].

**Fig. 1** FTIR-ATR spectra of the chia and flax mucilages obtained



### 3.3 Elaboration of the mock-ups

The mock-ups have been prepared taken into account in which purposes this kind of substances could be more useful in the field of conservation of cultural heritage.

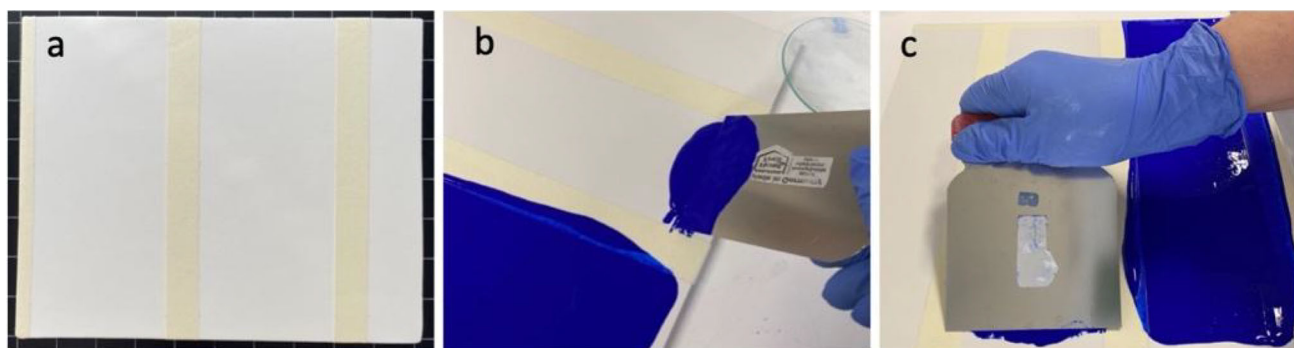
It is well known that one of the most important difficulties in conservation of old tempera paintings, especially when the medium is animal glue, either applied on canvas or wood, is to consolidate these sensitive layers. The product chosen should be effective to agglutinate the pigments, but also it must respect the colour and the brightness of the pictorial layers. The different consolidants that are usually used for this task, with better or worse results as some studies show, can be of synthetic origin, such as Paraloid® B72, Isinglass®, Aquazol® 200, or of natural origin, such as funori [23]. In many cases, one of the most important problems caused by the use of some of these products is about the gloss and colour changes that they produce in the paint films. This study tries to check whether the chosen vegetable mucilages could be a better alternative to the current products used. To do so, the authors have chosen a wooden support, in order to get a flat and hard surface which eases the colour and gloss measurement, but a canvas could be also used.

On the other side, the authors also wanted to know if the mucilages could be at least as useful as other products used as thickeners in the formulation of gels (like agar–agar or Laponite®) still employed to remove water adhesives of the reverses of art works. Sometimes, when a lined painting on canvas is restored, it is necessary to eliminate the canvas used to reinforce the original one and, after that, also the rests of adhesive (*gacha*) employed to adhere both cloths. On the other hand, a piece of cloth with rabbit-skin glue is sometimes used to patch tears in the textile support. If it is necessary to eliminate the patch for some reasons—because it is not useful anymore or even because it may be deforming the canvas—it is imperative to eliminate also the rests of the adhesive afterwards and to leave the cloth clean before restoring it. In the elimination of adhesives, it can be stated that the use of gels allows to apply controlled humidity, so that the possibility that the canvas and even the ground of the painting could be too wet, would be reduced. They can also avoid the use of a wet cotton swab, which can cause a deformation in the canvas by rubbing it. In this way, the other purpose of this work is to test if the mucilages obtained from flax and chia could be suitable for these tasks.

One mock-up was prepared to test the mucilages effectiveness as consolidants for rabbit-skin glue tempera painting (Mock-up A) and two more in the elaboration of hydrogels to eliminate natural adhesives (Mock-up BI and BII), as summarised in Table 1. For the purposes of these first tests, mock-ups were prepared trying to reproduce deteriorated artworks. Since this work includes the first results obtained, the authors have tried on mock-ups directly, without being aged. Nevertheless, we plan to test mock-ups subjected to artificial ageing in the next steps of this research.

The first mock-up (Mock-up A) was elaborated with the intention to imitate a disintegrated tempera painting. A plywood board was used as a support, on which it was first applied a layer of rabbit-skin glue previously hydrated in an adhesive: water ratio of 1:12 (w/w). This layer forms the link between the plywood substrate and the subsequent layers of the ground. Once the adhesive coat dried, the ground was applied, with the same glue and with gilder gypsum. To prepare the ground, 150 ml of the same solution of glue and water were mixed with enough amount of gilder gypsum using a glass rod.

In order to achieve the effect of poorly bound tempera pictorial layer, the rabbit-skin glue was prepared with lower concentration than usual. Therefore, a glue concentration to 1% in water (w/w) was used to agglutinate the ultramarine blue pigment that was applied on the plywood board.



**Fig. 2** **a** Plywood board prepared with ground and the overlapping masking tapes adhered to prepare the paint layers, **b** deposit of the tempera on the board and **c** spreading it with a wide flat spatula

**Table 3** Concentration of mucilages, their viscosity and decision about the convenience of being tested as consolidants

	Mucilage						
	Chia			Flax			
Concentration of mucilages(wt.%)	2%	1%	0.5%	2%	1%	0.5%	0.2%
Viscosity to be used as consolidant	High	Optimal	Optimal	High	High	High	Optimal
Tested on the mock-up	NO	YES	YES	NO	NO	YES	YES

Paint layers of around 130  $\mu\text{m}$  thickness were produced by spreading the tempera between two strips of masking tape using a flat spatula (Fig. 2). Finally, before the tempera was dried, the masking tape was removed, leaving as a result 24  $\times$  10 cm areas of blue rabbit-skin glue tempera.

On the other hand, to test the efficacy of mucilages in the elimination of natural adhesives, two mock-ups were made with Velázquez type linen canvas placed on two 22  $\times$  27 cm stretchers, (Mock-up BI and Mock-up BII). To fix the canvases to their wooden bars, staples were used.

On one of them (Mock-up BI), a solution of rabbit-skin glue, hydrated in a proportion of 1:12 at 50  $^{\circ}\text{C}$  (w/w), was applied by impregnation with a brush, in two coats.

For the other one, Mock-up BII, *gacha* was prepared according to a common recipe used in Spain. To make it, first it was necessary to prepare the Italian *colletta*, for which 3 kg of strong animal glue were hydrated in 2 l of water during 24 h. Afterwards, the excess of water was removed, and this glue was heated until it was dissolved. Afterwards, the rest of the components was added (vinegar 2 l, ox gall 50 g). Once the mixture was homogeneous, it was poured into trays, 3 cm thick, and left to cool. Once it had gelled, it was cut into small prisms and laid out on a flat surface to dry, turning them for several days, so they would dry properly on all sides. Once the *colletta* was dried, the *gacha* was prepared. To do so, 300 g of flour were added to 1 l of water (23  $^{\circ}\text{C}$ ) placed in a pot. The mixture was cooked in a bain-marie, and it was beaten with a hand blender to remove any lumps. After that, it was continuously stirred with a wooden spoon. Then, 100 g of *colletta*, weighed dry and then hydrated, were put to melt in a bain-marie, and 37 g of Venice turpentine were added. Then, this mixture is added to the pot with the water and flour, stirring constantly. Once the *gacha* was prepared in this way, two coats of it were applied at 50  $^{\circ}\text{C}$  by brush impregnation over the canvas.

The adhesives in both mock-ups were left to dry before testing the mucilages.

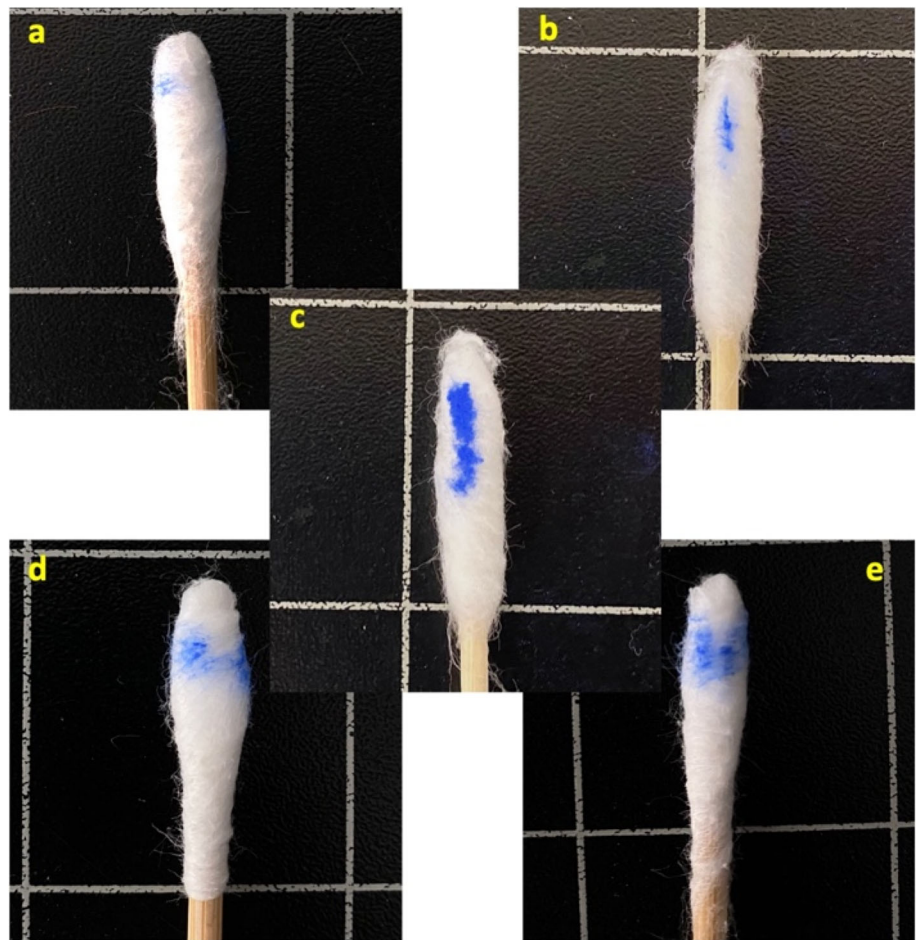
### 3.4 Testing of mucilages on mock-ups: consolidants and cleaning gels

The first step was to hydrate the mucilages extracted from the seeds. To evaluate their use as consolidants, different concentrations were tested, as shown in Table 3. The viscosity of the consolidant must be low enough to penetrate the paint film, but not so low that it penetrates too deeply and does not remain in the thickness of the pictorial layer.

Flax mucilages show higher viscosity than the chia ones hydrated to the same concentration. For this reason, flax mucilage was tested also in a lower concentration (0.2%).

After hydrating the mucilage for 24 h, the mixture was heated at 50  $^{\circ}\text{C}$  for approximately 10 min with a constant stirring at 600 rpm, to facilitate the dissolution of chia and flax mucilages. This temperature was selected with the aim of applying the minimum heat needed to the mixture to avoid possible alterations associated with high temperature. Finally, the mucilages were allowed to cool at room temperature. In the case of the 0.5% flax mucilage, one part was allowed to cool until 40  $^{\circ}\text{C}$  and the other to 23  $^{\circ}\text{C}$  (room temperature), to test the effect of the increased temperature in the consolidation process and the possible dissolution of the tempera.

**Fig. 3** Swabs: **a** treated area with 0.5% flax mucilage, **b** treated area with 0.2% flax mucilage, **c** untreated area, **d** treated area with 1% chia mucilage and **e** treated area with 0.5% chia mucilage



The application method consisted on placing a Japanese paper (*Productos de conservación*, ref. 25,501, 6 g) on the surface of the tempera painting to be consolidated and to apply the mucilages with a flat and soft brush. The placement of the Japanese paper between the tempera and the mucilage was intended to avoid the dilution of the paint during the application of the consolidant. Three layers of consolidant were applied consecutively. Immediately after applying the third layer, the Japanese paper was gently removed to prevent it from adhering.

To test the efficacy of mucilages in the production of cleaning gels, mucilages from chia and flax were first hydrated in a mucilage/distilled water ratio of 1:10 (w/w) during 24 h at room temperature (23 °C) to obtain a homogeneous gel. After 24 h, the mucilages were tested, following the methodology described below.

A small amount of the mucilages were placed on selected areas, and they were left to act for 10 and 20 min. Afterwards, the mucilages were removed using a soft plastic spatula. Finally, the area was left to dry.

#### 4 Results and discussion

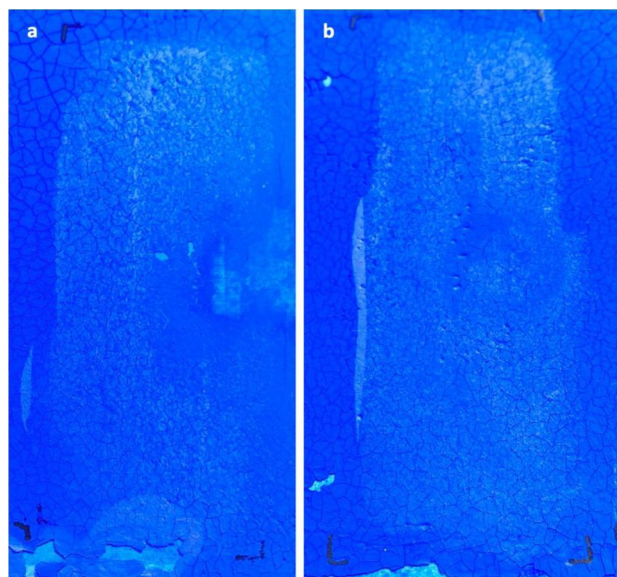
To test the efficacy of mucilages as consolidants on Mock-up A, dry cotton swabs were gently rubbed on the surfaces of the paint films before and after consolidation (Fig. 3). In the case of the flax mucilage, it was able to consolidate the pictorial film, with both concentrations tested, 0.5% and 0.2%, and with both temperatures, 23 and 40 °C, as show the nearly clean cotton swabs. In contrast, the efficacy of chia as consolidant was much lower than flax mucilage, since the swabs rubbed in both areas where the mucilage was applied at 1% and at 0.5% appeared still rather stained (Fig. 3).

The effect of the temperature during the consolidation process was tested using flax mucilage at 0.5% concentration applied at 23 and 40 °C. When the temperature is 40 °C, a high amount of pigment is attached to the Japanese paper, as can be seen in Fig. 4, whilst a substantially smaller amount of blue pigment remains in the paper after the application of both mucilages, chia (1% and 0.5%) and flax (0.5% and 0.2%) at room temperature (23 °C). As a result, it can be stated that the application temperature is a factor to be considered when evaluating the suitability of the application methodology to consolidate tempera painting: the higher the temperature, the faster the tempera is dissolved, making the pigment to adhere to the paper during the consolidant application.

**Fig. 4** Traces of pictorial film on the Japanese paper interposed when flax mucilage at 0.5% was applied. The upper paper corresponds to the one applied at 40 °C and the lower to the one applied at 23 °C



**Fig. 5** Whitish veil formed where chia mucilage was applied at **a** 0,5% and **b** 1%



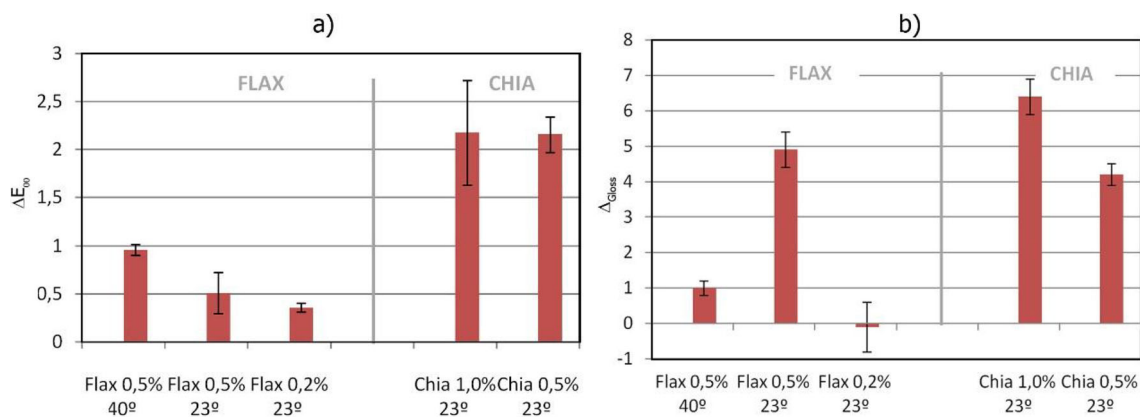
Observation of paint films after consolidation evidenced a whitish veil, visible to the naked eye, formed in all areas where the chia mucilage was applied at both 1 and 0.5% concentration (Fig. 5). This assessment is corroborated with the measurements of colour variation. Thus, an increase in the colour parameters of ( $\Delta E_{00}$   $2.18 \pm 0.54$ ;  $\Delta E_{00}$   $2.16 \pm 0.18$ ) and gloss to 85° ( $\Delta_{\text{Gloss}}$   $6.4 \pm 0.5$ ;  $\Delta_{\text{Gloss}}$   $4.2 \pm 0.3$ ) is experienced for the 1 and 0.5% concentrations, respectively. On the other hand, the use of flax mucilage did not produce a whitish veil visible to the naked eye, and only a significant variation in gloss occurs when applied at 0.5% ( $\Delta_{\text{Gloss}}$   $4.9 \pm 0.5$ ), but colour changes are negligible ( $\Delta E_{00} < 1$ ), no matter the concentration used (Fig. 6).

To study the effectiveness of the mucilages as cleaning gels, Mock-ups BI and BII were analysed with FTIR-ATR and observed with direct and raking natural lights after cleaning.

Reference FTIR-ATR spectra of the linen canvas support, the rabbit-skin glue, the *gacha*, the linen canvas support with rabbit-skin glue and the linen canvas support with *gacha* (Fig. 7), in addition to chia and flax mucilages (Fig. 1), are used to evaluate the surfaces cleaned with the different hydrogels.

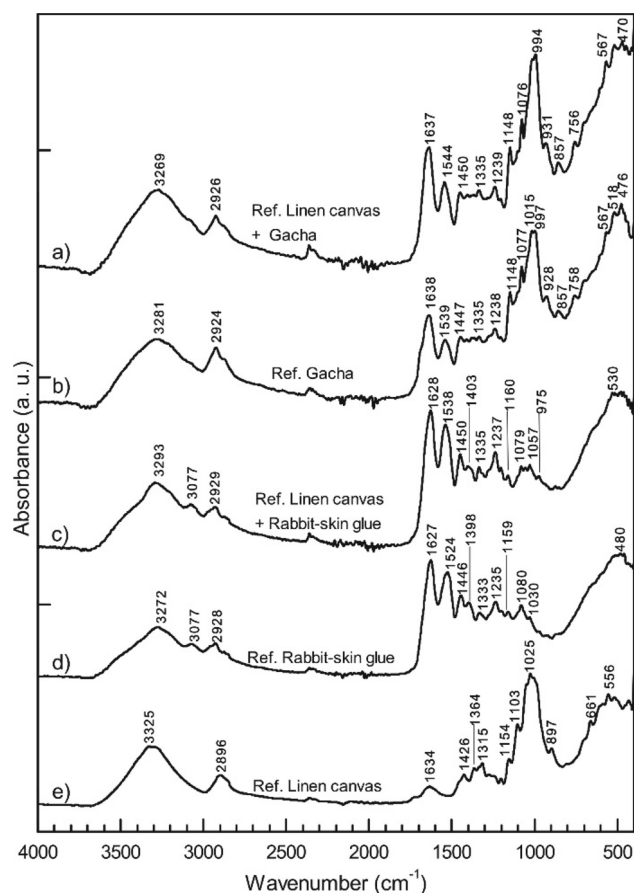
For the evaluation of the cleaning process, it has been assumed that a greater cleaning effectiveness will mean more intense bands corresponding to the linen canvas, whilst less cleaning means more intense bands of the rabbit-skin glue in Mock-up BI or *gacha* in Mock-up BII.

The significant bands of both compounds which did not show strong overlap were chosen. Thus, the strong bands of the rabbit-skin glue/*gacha* corresponding to the amide I group and amide II group ( $1627/1638 \text{ cm}^{-1}$  and  $1524/1539 \text{ cm}^{-1}$ , respectively) or linen



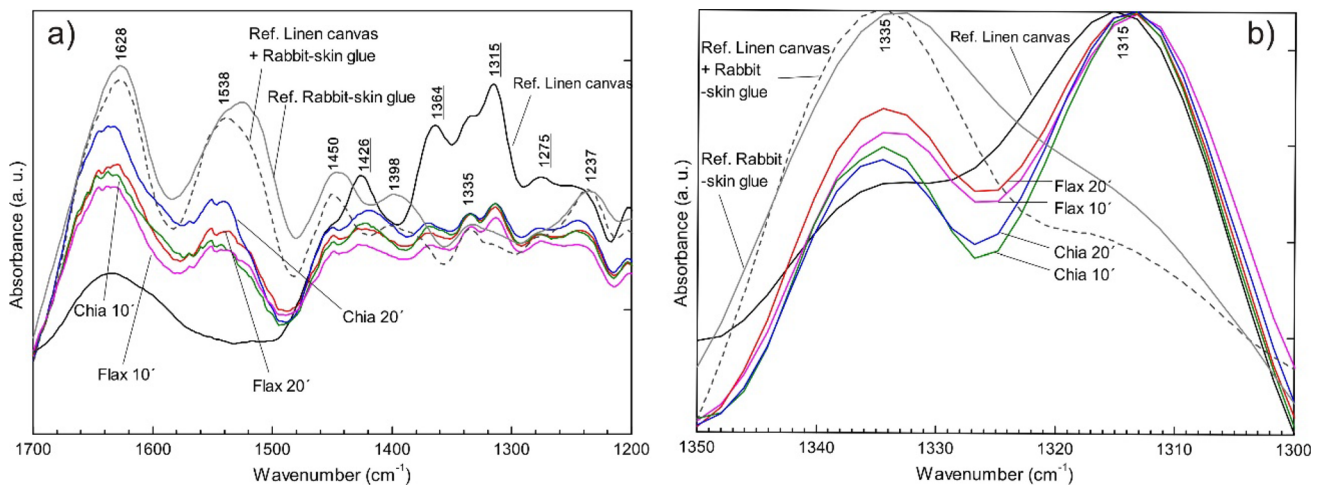
**Fig. 6** a Variations of colour and b gloss with the application of the mucilages tested as consolidants

**Fig. 7** FTIR-ATR reference spectra a Linen canvas with *gacha*, b *gacha*, c linen canvas with rabbit-skin glue, d rabbit-skin glue and e linen canvas

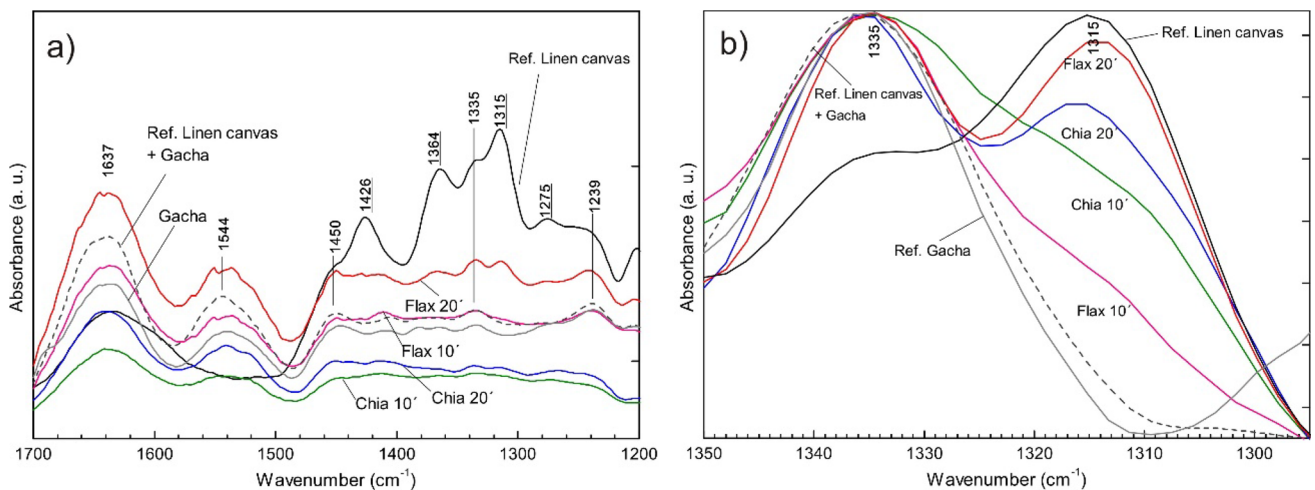


canvas bands corresponding to carbohydrates (1100–900  $\text{cm}^{-1}$ ) could not be considered, since they overlap with the bands of the mucilages, chia and flax (Fig. 1 and Fig. 8a). Consequently, it was necessary to consider other significant secondary bands: the band at 1335  $\text{cm}^{-1}$ , attributed predominantly to the  $\text{CH}_2$  wagging vibration in the rabbit-skin glue and *gacha* [24, 25] and the band at 1315  $\text{cm}^{-1}$ , assigned to C–O–H and H–C–C bending vibrations to the linen canvas [26]. Figure 8b shows these bands, normalised to the band at 1315  $\text{cm}^{-1}$ , of the linen canvas alone and the mock-up BI (with rabbit-skin glue) before and after being cleaned with both mucilages. Spectra of the rabbit-skin glue alone are added as reference. As can be noticed, the cleaning treatments with both flax and chia mucilages produce a drastic decrease in the band at 1335  $\text{cm}^{-1}$  proving their efficacy in the elimination of the rabbit-skin glue. Moreover, it can be established that the chia mucilage was able to eliminate the glue slightly better, independently of the application time.





**Fig. 8** **a** FTIR-ATR spectra of the rabbit-skin glue surfaces cleaned with the different hydrogels and of the references of linen canvas with rabbit glue adhesive and linen canvas, in the range of  $1700\text{--}1200\text{ cm}^{-1}$ ; **b** spectra in the  $1350\text{--}1300\text{ cm}^{-1}$  region fitted over the  $1315\text{ cm}^{-1}$  band.



**Fig. 9** **a** FTIR-ATR spectra of the *gacha* surfaces cleaned with the different hydrogels and of the references of linen canvas with *gacha* adhesive and linen canvas, in the range of  $1700\text{--}1200\text{ cm}^{-1}$ ; **b** spectra in the  $1350\text{--}1300\text{ cm}^{-1}$  region fitted over the  $1335\text{ cm}^{-1}$  band.

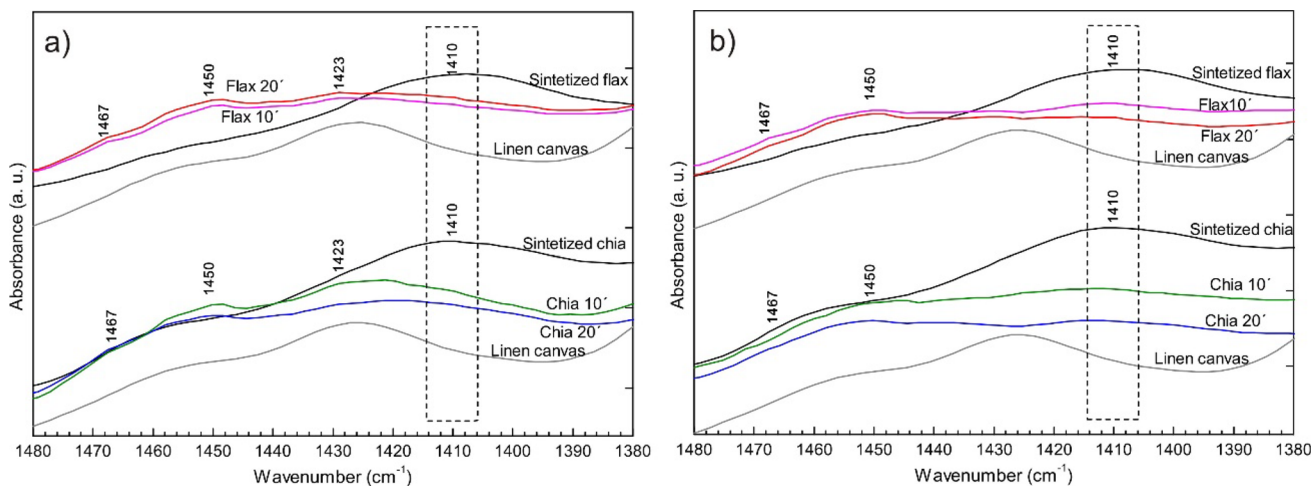
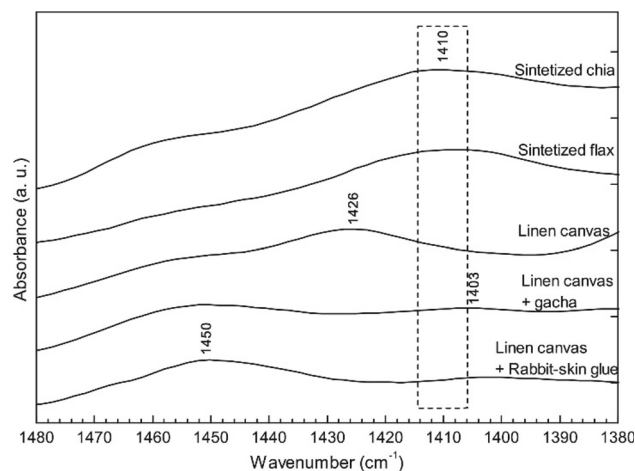
For the evaluation by FTIR-ATR of the *gacha* surfaces cleaned with the different hydrogels (Mock-up BII), as in the Mock-up BI, the reference spectra were used (Fig. 9a). As in the previous case, the band at  $1335\text{ cm}^{-1}$  from the linen canvas with *gacha* was compared to the band at  $1315\text{ cm}^{-1}$  of the linen canvas. In this case, the spectra were normalised to the band at  $1335\text{ cm}^{-1}$  related to the presence of *gacha*, since the band of the support is of lower intensity due to the covering power of the *gacha* compared to the rabbit-skin glue, so it can be considered that the increase in intensity of the band at  $1315\text{ cm}^{-1}$ , related to the presence of linen canvas, implies a greater removal of *gacha* during the cleaning procedure. In this case, the application of flax mucilage for 20 min produced the best result since the band of the linen canvas is more intense. Application of chia hydrogel for 20 min was also able to clean enough to see the support, whilst an application time of 10 min was not enough in any case.

For the determination of possible mucilage residues, it has been considered the presence of the low intensity broad band at  $1410\text{ cm}^{-1}$ , common to chia and flax mucilage and corresponding to symmetrical C-OO stretching. This band does not overlap with other bands of the reference patterns, although it is close to the band  $1403\text{ cm}^{-1}$  of adhesive rabbit-skin glue and *gacha* (Fig. 10).

All the FTIR-ATR spectra of the rabbit-skin glue surfaces cleaned (Mock-up BI) with the different hydrogels show a very small shoulder at  $1410\text{ cm}^{-1}$ , which may indicate the existence of a small amount of chia and flax mucilage residues (Fig. 11a). In the same way, the FTIR-ATR spectra of the *gacha* surfaces cleaned with the different hydrogels and application times (Mock-up BII) show the presence of a small shoulder at  $1410\text{ cm}^{-1}$  as well, which also indicates the possible presence of chia and flax mucilage residues, being slightly higher when they were applied for 10 min (Fig. 11b).

A visual examination of the Mock-up BI with direct light (Fig. 12) revealed that the chia applied for 20 min generated a dark halo around the area (Fig. 12 d), which was not observed after 10 min (Fig. 12 b) nor in the areas where flax mucilage was applied. Under raking natural light, it could be appreciated that all the zones where the mucilages had been applied and had matt appearance,

**Fig. 10** FTIR-ATR spectra of the chia and flax extracted mucilages, linen canvas, line canvas with *gacha* and linen canvas with rabbit-skin glue, in the range of 1480–1380  $\text{cm}^{-1}$ .



**Fig. 11** FTIR-ATR spectra of the surfaces with the different hydrogels in the range of 1480–1380  $\text{cm}^{-1}$ : **a** rabbit-skin glue surfaces cleaned; **b** *gacha* surfaces cleaned

which indicates the elimination of the adhesive, in contrast to the untreated areas, which had shiny appearance due to the presence of rabbit-skin glue (Fig. 12d1).

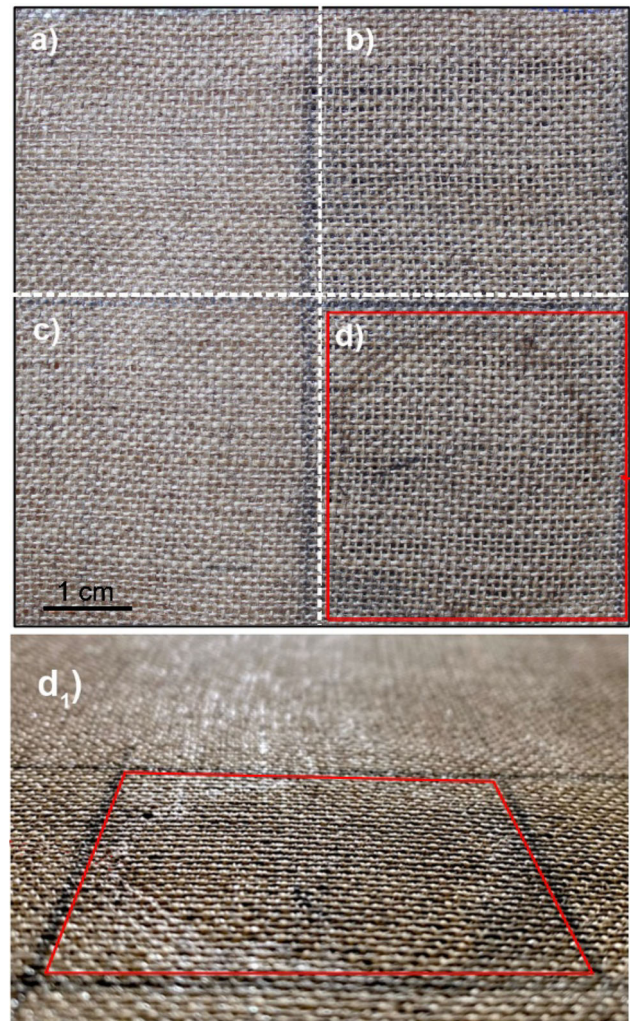
Visual observation of the treated areas in Mock-up BII shows that the application of the hydrogels produced a change in appearance due to the elimination of the adhesive and the canvas underneath could be observed (Fig. 13). Images obtained with the microscope using UV light (Fig. 13) showed that, after the application of flax and chia mucilages for 10 min, some clusters of *gacha* still remain, seen as white areas in the images, whilst when the application time increases to 20 min, both mucilages satisfactorily eliminated the *gacha*. However, in the case of chia, the surface of the canvas was slightly deformed (Fig. 13).

## 5 Conclusions

It can be stated that, under the conditions tested and with the procedure used here, flax mucilage does have the ability to consolidate disintegrated layers of rabbit-skin glue tempera producing minimum colour and gloss changes, whereas chia has less efficacy to consolidate the tempera and produces a whitish veil visible to the naked eye.

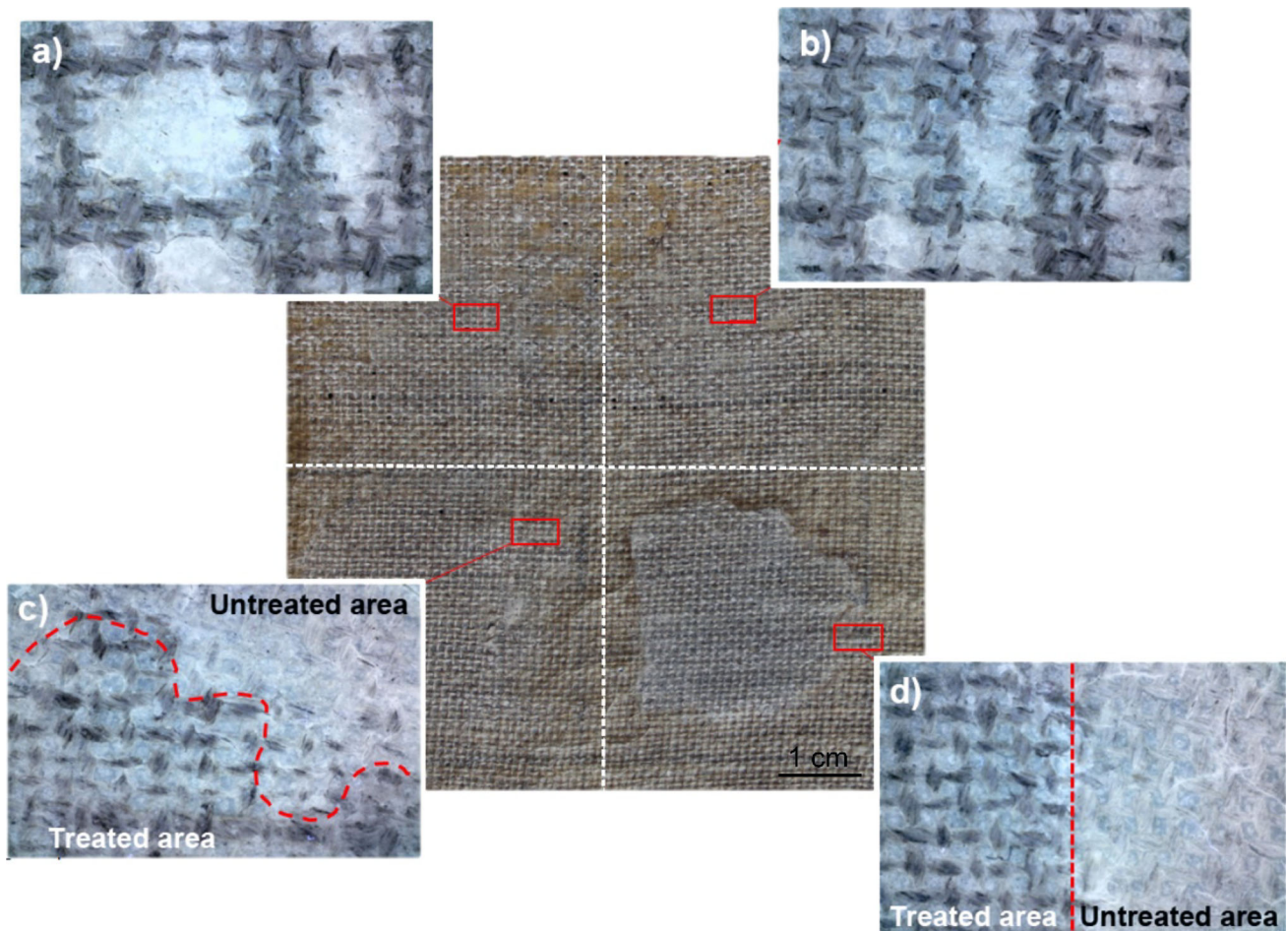
About the efficacy of mucilages used as thickeners in the production of hydrogels, chia and flax mucilages had the ability to gel the aqueous phase. The hydrogel with chia was able to release more of the aqueous phase after 20 min of action, allowing a greater removal of the adhesive, but with the disadvantage of producing a deformation of the canvas and a dark halo. Therefore, it should probably be used for less time than flax at the concentrations tested. In all cases, it has been observed that traces of adhesives remain after cleaning.

**Fig. 12** Final appearance of the Mock-up B1 with rabbit-skin glue observed with direct light after the application of: **a** Flax mucilage for 10', **b** chia mucilage for 10', **c** flax mucilage for 20', **d** chia mucilage for 20' and **d<sub>1</sub>**) this one under raking light



According to the approach made with this research, the main limitation of this methodology is that the mock-ups used were not subjected to artificial ageing, so it is possible that the results may change slightly. Variables such as action times are affected, in the case of hydrogels, since it is possible that in older adhesive layers, these may be longer, as these will be more rigid, and their removal could be more difficult. So, in the future, it is planned the use of aged mock-ups, to simulate old cultural heritage.

After the use of both chia and flax mucilages for the elimination of natural adhesives, small amounts of residues have been detected, which makes it necessary to carry out accelerated artificial aging tests to determine the long-term behaviour of the mucilages, in terms of their stability, chromatic variation and binding power. These tests could show if the mucilage residues will affect some aspect of the pictorial layers or supports.



**Fig. 13** General image of the area of *gacha* Mock-up BII, where flax and chia mucilages were applied and detail images under 35x, using UV light. **a** flax and **b** chia, in both cases left to act for 10' (images taken from the centre of the treated area); **c** flax and **d** chia that were left to act for 20' (images taken in the limit between the treated and the untreated areas)

Finally, the authors are also interested in the research of other applications of mucilages in the field of conservation of heritage. On the one hand, as thickeners in the elaboration of hydrogels used to clean sensitive pictorial layers in different supports. On the other hand, as consolidants, using other methods to apply them and testing them on other kinds of tempera painting, such as egg or casein.

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## Declarations

**Conflict of interest** All authors certify that they have no affiliations with or involvement in any organisation or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

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